Influence of Al_2O_3 Additive on Mechanical Properties of Wollastonite Glass-Ceramics

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Abstract: In this paper, wollastonite glass-ceramics and composites of wollastonite glass– ceramics with 2.5, 5, 10 and 20 weight percent alumina with an average size of 2 microns and also wollastonite glass-ceramics with 2.5, 5, and 10 percent alumina with an average size of 40 nanometers were produced without pressure by Sintering and their physical properties (e.g. bulky density, the percentage of linear shrinkage and relative density) were measured. Sinter operation in the temperature range of 1030-1170 °C was performed for 3 hours. Existing phases in composites by X-ray Diffraction (XRD) and their structure were examined by Scanning Electron Microscopy (SEM) and while measuring mechanical properties of composites such as flexural strength, hardness and compared fracture toughness with base glass ceramic was performed. Results indicate that adding 2.5 percent micron-sized alumina to wollastonite glass-ceramics decreases the flexural strength from 8.01±120 to 10.26±50 MPa and its fracture toughness declines to 0.8±0.74, while by adding 2.5 percent nano-alumina to wollastonite glass – ceramics, the flexural strength increases from 8.01±120 to 20.7±133 MPa and its fracture toughness improves up to 1.40±10.

Keywords: Glass-Ceramic Composite, Nano-Alumina, Mechanical Properties, Sintering, Thermal Properties, Wollastonite

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1 INTRODUCTION

Reinforcing glass-ceramic with other materials such as metals and ceramics composites can be made to improve the properties of the original matrix. This practice is generally done to improve the low fracture toughness of glass and glass–ceramics. Historically, the first laboratory-scale research projects have been carried out for the past 40 years. In ceramics, it is possible that the crystallization or precipitation of one phase in the other phase creates multi-phase and multi-component structure which are called composite [1]. Al_2O3 composites because of some extensive properties such as high hardness, low electrical conductivity, good chemical stability and oxidation resistance have been studied widely. Most studies are focused on dispersing Al_2O_3components of the composite to improve the mechanical properties like tensile and tear strength [2-3]. Glass-ceramics owing to properties such as low density and low cost are important engineering materials. Various processes can employ build glass - ceramics composite. Raw body (which is actually a mixture of glass powder and strengthening particles) can be produced in several ways such as uniaxial pressing, cold isostatic pressing or slurry casting. The internal stresses in composite field depend on the difference in coefficients of thermal expansion, elastic properties of field and reinforcing particles [4]. Glass–ceramics of the SiO2-CaO-MgO system are important materials because of their special durability and mechanical properties which arise from precipitation of diopside and wollastonite [5]. Uneven thermal contraction of field and the second phase during cooling Sintered composite result in residual stresses in them. If the thermal expansion coefficient of reinforcing phase is larger than the thermal expansion coefficient of the field, residual stress in field will include radial tensile and ambient pressure and second phase components will be under ambient tensile. On the other hand, when the thermal expansion coefficient of reinforcing phase is less than thermal expansion coefficient of matrix, there will be ambient tensile and radial pressure in the field and radial tensile in reinforcing phase [6]. By selecting the proper ratio of the second phase and field, the type and amount of residual stress can be controlled in such a way that several toughening mechanisms can be provided [1]. Owing to the fact that aluminum titanate Al_2TiO_5 (AT) has minimal heat expansion, high thermal shock resistance, and high melting point, it has special feature as an interesting ceramic. Research suggests that combination of aluminum composite with other materials increases resistance to thermal and mechanical shock. It is also said that in composites of aluminum titanate-diopside, thermal shock has been improved. In addition, a variety of metallic and ceramic materials can be used as a booster. With increase in amount of aluminum titanate to diopside, there is no guarantee of a steady flow of the glass phase during sintering. This procedure increases the firing temperature and the maximum shrinkage. It seems that Aluminum titanate particles in glass-ceramics context, because of the high sintering temperature and cooling rate improve micro cracks. Start of the effect of hydrostatic pressure causes the closure of micro cracks; therefore, according to the properties of aluminum titanate in micro-cracks and its strong impact on high modulus, it increases flexibility in the context of glass - ceramics and composite strength and stiffness but reduces ductility and fracture threshold. Radial cracks around particles of aluminum titanate and raising level of porosity are other important factors to reduce ductility threshold. Adding aluminum titanate to the glass-ceramics diopside product more than ten percent can improve the mechanical properties [5]. In another study, composite Al_2O_3-ZrO_2 (Y_2O_3) which has good resistance was examined. The subject of this study was the flexural strength and stiffness of Al_2O_3 [7-8]. We have recently developed a reduction method of converting Ag nanospheres and antibacterial activity [9], preparation of Ag/ZnO nanocomposite [10] synthesis and comparison nanosilver particles and nanosilver plates for the oxidation of ascorbic acid [11-12]. E. Yekta and et al. have studied the effect of adding different amounts of TiO_2, CaF2 and ZrO_2 oxides on sintering and crystallization behavior of CaO- Al_2O_3-SiO_2 glass system. Results of this study showed that the addition of CaF_2 can improve sintering and crystallization because of lower viscosity of glass systems while by Al_2O_3 additives, crystallization peak temperature can have the shift to higher temperatures and increase Sintering temperature for compressibility but do not affect improvement of mentioned properties owing to the increased viscosity [13]. In addition to the above research, substitution effect of Al_2O_3 by ZnO on the sintering and crystallization behavior of glass - ceramics of MgO- Al_2O_3-SiO_2 system can be studied [14].

2 MATERIALS AND METHODS

2.1. Sample Preparation

The raw materials for wollastonite glass-ceramic production in this study according to "Table 1" are Silica, Calcium Carbonate, Sodium Carbonate, Iron Oxide and Tungsten Oxide with high purity from Merck, Germany Company.

Table 1 Chemical composition and glass components

Components	SiO_2	CaO	Na_2O	Fe_2O_3	WO_3
Wt %))	59.68	27.25	5.08	6	2

Also Al_2O_3 with particle size of 2 μm and size of 40 nm and 99 percent purity from Martinzwerk Germany was used as a reinforcing factor in composites. The raw materials were thoroughly mixed and matched. Then, the mixture was concentrated under pressure inside alumina crucibles and in the oven with temperature of 1410 °C, and in atmospheric environment was thawed. Retention time of molten glasses was in melting temperature of approximately thirty minutes. The molten glass is poured into metal molds and for annealing in ATASH1500 EXCITON furnaces were heat treated at a temperature of 580 °C for 1 hour and other molten glass quickly was cooled in deionized distilled water. Ferrite obtained from each of glasses first by hand alumina mortar and then inside the electric pestle was milled for one hour. After that, it was milled wet inside fast mill with pellets and alumina mortar ethanol for 15 min. Then, all the fine powders were then deposited through sifting with a grain size of 38 microns in slurry method for 24-48 hours. After drying in the dryer at temperature of 100 °C, forming powder was obtained. Particle size distribution and mean particle size of the glass powders and alumina particles were examined by particle aggregating device. Reaching maximum density, the lowest percentage of water absorption and linear shrinkage, are measures for determining the optimum temperature of Sinter. Thermal treatments in all cases, increasing the temperature from room temperature to 450 °C with a heating rate of 5 °C/min, and maintenance at this temperature for one hour to lose organic volatiles were carried out on the samples. Samples were then cured in an electric furnace with a heating rate of 5 °C/min at a maximum temperature of 1700 °C. Retention time was three hours and cooling rate was chosen 5 °C/min. Sintering evaluation of each compound with a linear shrinkage measurement was reviewed by using a caliper with an accuracy of 0.05 mm. Bulk density of the sintered samples was determined by the Archimedes method. The real density was measured by Pycnometery according to ASTM D2320-98 standard.

2.2. Preparation of Composites

Glass powder with 2 μm alumina in a weight ratio of 2.5, 5, 10, 20 is mixed with α- Al_2O_3 (40 nm) in ratio of 5 and 10 weight percentage. The process of mixing glass powders with alumina particles was carried out in fast mill for 15 min in ethanol. After drying the mixture at a temperature 100 °C/min in the dryer, forming powders were prepared. For naming glass - ceramics resulted from heat treatment and also for brevity, Contract Marks were used. In this naming, "W" was used to represent wollastonite glass-ceramic composition and "A", for micron-sized Al_2O_3 reinforced additive as well as "A" for Nano-sized α- Al_2O_3 additive. Glass-ceramic composites containing 2.5, 5, 10 and 20 wt% α- Al_2O_3 in micron size respectively were named "$WA_{2/5}$, WA_5, WA_{10} and WA_{20}" and glass-ceramic composites containing 2.5, 5 and 10 wt% α- Al_2O_3 in nano size were named "$WAn_{2/5}$, WAn_5, and WAn_{10}", respectively [15].

2.3. Methods of Analysis

Particle size distribution and mean particle size of the glass powders and alumina particles were examined by particle aggregating device. To determine the phase composition of the glass-ceramics and prepared composites, first sintered tablets were converted to powders inside electric agate mortar and then were passed through a sieve with a particle size of 45 microns. X-ray diffraction patterns using a Siemens X-ray machine (Siemens, Model D500) with light of 1533.0 nm wavelength and at different ranges (from 10 to 80 degrees) were applied. Identifying patterns were done by using JCPDS reference card and X-Pert software. Reviews on microstructure of samples were done using Scanning Electron Microscope (SEM, Philips XL30) equipped with AX ED analysis.

Samples were prepared for microscopic evaluation by polishing with 200, 400, 600, 800, 1000, 1200, and 1500 sandpaper, and 1-micron diamond paste. After polishing operations, samples were cleaned with ultrasonic device and were chemically etched for 45 seconds with a solution of HF 2% hydrofluoric acid. Hardness evaluation of the glass–ceramics' samples and Sintered and fully polished composites, was performed by using a Vickers micro hardness tester (Buehler, Micrometre I) under a load of 100 g at 30 seconds.

In order to measure the three-point flexural strength and toughness, samples with dimensions of 25 × 5 × 6 mm3 at optimum temperature of 1030-1100 °C as tablets' conditions were sintered. The flexural strength of sintered and polished Samples was measured by three-point method with a distance of 15 mm between the abutments. The number of samples tested for each compound was seven and measurement was performed in a jaw speed of 0.5 mm/min in an ambient atmosphere. Fracture toughness of the samples was measured by SENB method and in the three-point bending mode with a distance of 15 mm between the fulcrums. Sample dimensions B=6, W=5 and S=15 mm were selected. A groove with a depth of approximately a=2.5 mm by low-speed micro-cutting device (Buehler, Isomet) in polished samples were established. Groove depth was measured with a calibrated optical microscope. Nominal width of created groove was 0.3 mm. The number of samples tested for each compounds was seven and measurement was performed in a jaw speed of 0.5 mm/min in an ambient atmosphere. Toughness was performed according to ASTM E399 standard [16-17].

3 RESULTS AND DISCUSSION

3.1. Sintering and Crystallization

After sintering, according to the previous method, the linear shrinkage, bulk density and percent relative density of composites were measured. Measured linear shrinkage for composites $WA_{2.5}$ and WA_5 and WA_{10}, was 12.44, 10.6 and 10.1 percent, respectively. Trend of changes in linear shrinkage of composites is similar to trend of glass–ceramics: By increasing temperature, first linear shrinkage increases, then declined to take a certain fixed amount. Also, increased alumina particles in composite samples reduce the amount of glass phase for sintering through the non-fluent flow and as well as linear contraction.

According to bulk density values ("Fig. 1"), optimum temperature of Sintering in $WA_{2.5}$, WA_5 and WA_{10}, respectively is 1030, 1075 and 1095 °C. Also, optimum temperature of WA_{20} is 1170 °C [18].

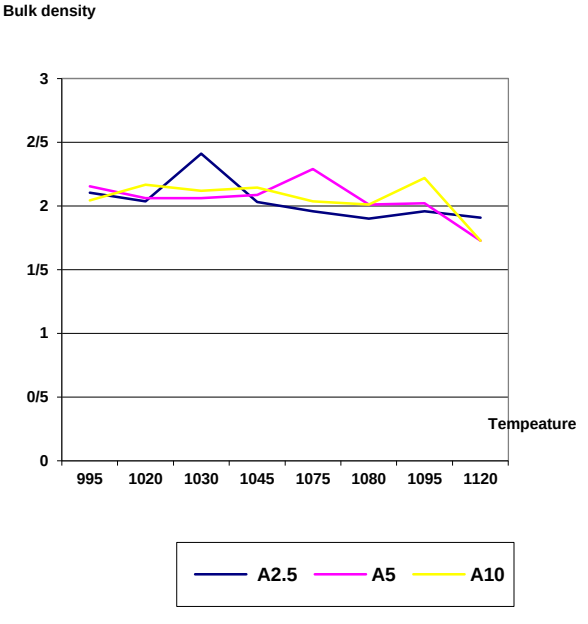

Fig. 1 Changes in bulk density of wollastonite composites (micron-sized alumina).

According to bulk density values ("Fig. 2"), optimum temperature of Sintering in $WAn_{2.5}$, WAn_5 and WAn_{10}, respectively is 1030, 1075 and 1090 °C. Sintering temperature of above components, compared to composite $WA_{2.5}$, WA_5 and WA_{10} has not changed much.

Percent relative density of all composites are listed in ("Table 1"). As can be seen, bulk density of all composites, compared to the bulk density of wollastonite glass– ceramics has fallen. Also, percent relative density of composite $WA_{2.5}$, WA_5 and WA_{10}, WA_{20} and $WAn_{2.5}$, WAn_5 and WAn_{10} composites, compared to the wollastonite glass–ceramics whose percent relative density is 95.37 has decreased. Although with adding the second component to wollastonite glass-ceramics, composite density is expected to increase but due to the porosity and non-complete sintering, the density has declined, so by increasing the percentage of second component in two micron and Nano groups, relative and bulk density have dropped. Meanwhile, bulk and relative density of composites with Nano component are more than relative density of composites with micron component [17].

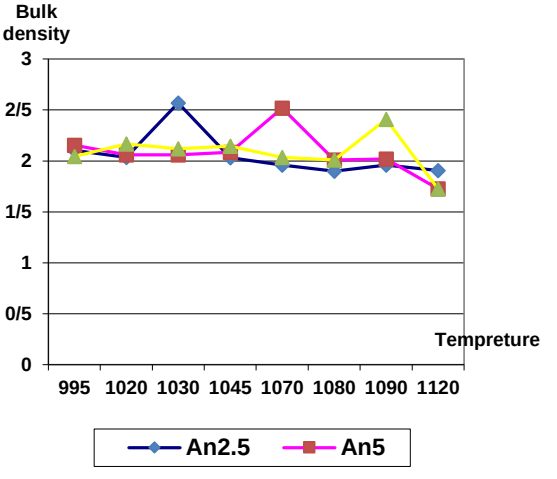

Fig. 2 Changes in bulk density of wollastonite composites (Nano-sized alumina).

3.2. Microstructure analysis

In "Fig. 3", X-ray diffraction patterns of $WA_{2.5}$, WA_5 and WA_{10}, and WA_{20} samples which respectively have heat treated at temperatures of 1030, 1075, 1095 and 1170 °C, have been shown. It is seen that $WA_{2.5}$ and WA_5 compounds have wollastonite and alumina phases and gradually by increasing sintering temperature of the samples in some cases, the type of formed phase has been changed, such that formation of Anorthite is evident from around 1095 °C onwards. WA_{10} has wollastonite, alumina and a little Anorthite phases and WA_{20} has Anorthite and alumina phases. In WA_{10} wollastonite has become a bit Anorthite, so peak intensity of wollastonite has slightly decreased.

Also, in $WA_{2.5}$, W_{A5} and WA_{10}, and WA20, it can be seen that by increasing percentage of second component i.e. α- Al_2O_3, peak intensity of the one with alumina has increased ("Fig. 3"). In "Fig. 4", X-ray diffraction patterns of $WAn_{2.5}$, WAn_5 and WAn_{10} samples, which respectively have heat treated at temperatures of 1030, 1075, and 1090 °C, have been shown. It is seen that $WAn_{2.5}$ and WAn_5 compounds have wollastonite and

alumina phases and Wan10 have wollastonite, alumina and a little Anorthite phases ("Table 2"). Also in $WAn_{2.5}$, WAn_5 and WAn_{10}, it can be seen that by increasing percentage of second component i.e. α- Al_2O_3, peak intensity of the one with alumina has increased. By comparing the phase analysis of $WA_{2.5}$ with $WAn_{2.5}$, WA_5 with WAn_5 and WA_{10} with WAn_{10}, we conclude that because of nanoparticles and 2 to 40 nm fine alumina particles we face wider peaks ("Fig. 4") [16].

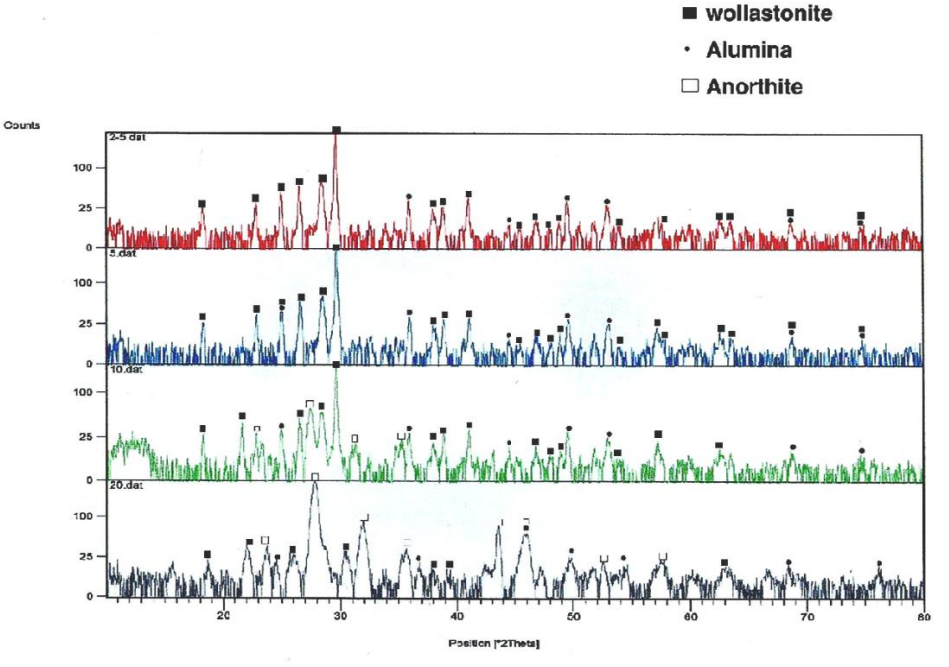

Fig. 3 X-ray diffraction patterns of $WA_{2.5}$, WA_5 and WA_{10}, and WA_{20} samples in optimum Sintering temperature.

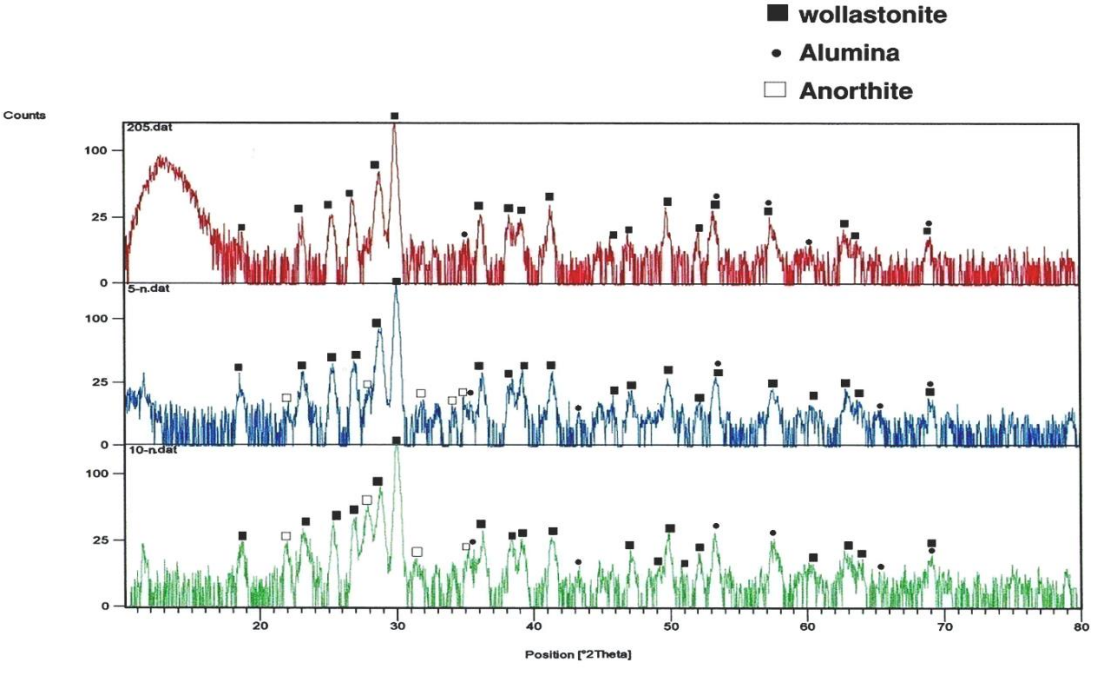

Fig. 4 X-ray diffraction patterns of $WAn_{2.5}$, WAn_5 and WAn_{10} samples in optimum sintering temperature.

Table 2 Composition of phases in specimens

Phases	composition
wollastonite	Glass – ceramic
Wollastonite and alumina	$WA_{2/5}$ and $WAn_{2/5}$
Wollastonite and alumina	WA_5 and WAn_5
Wollastonite, Alumina and Anorthite	WA_{10} and WAn_{10}
Wollastonite, Alumina and Anorthite	WA_{20}

3.3. Scanning Electron Microscope (SEM) Iimages

Scanning Electron Microscope (SEM) images are shown in "Figs. 5 to 9".

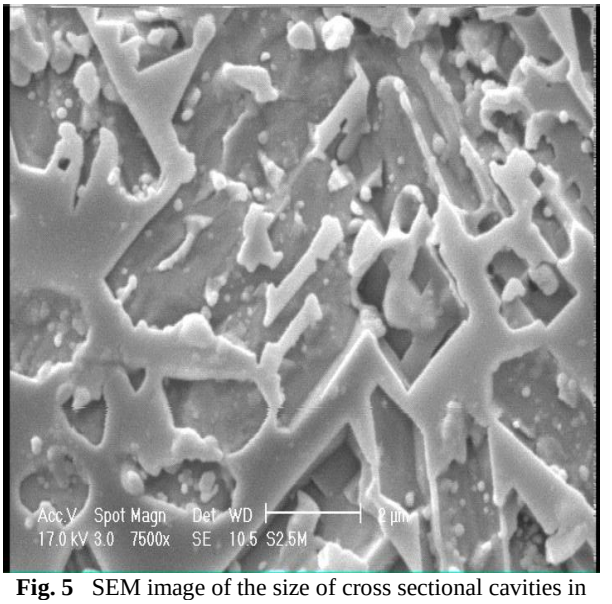

Fig. 5 SEM image of the size of cross sectional cavities in $WA_{2.5}$ (7500×).

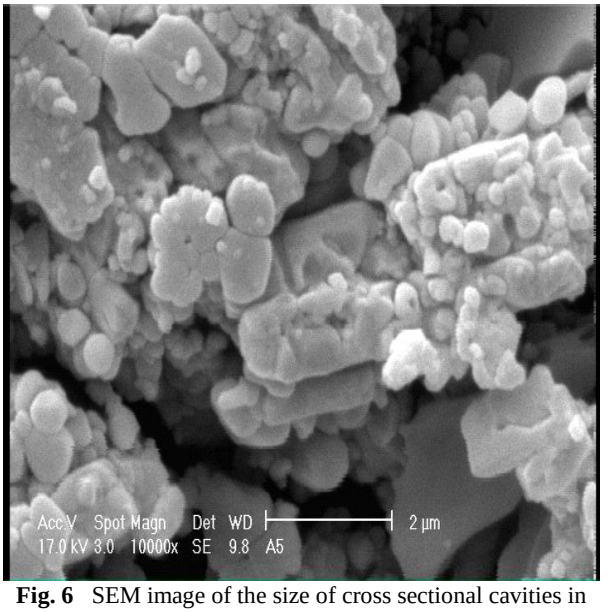

Fig. 6 SEM image of the size of cross sectional cavities in WA_5 (×10000).

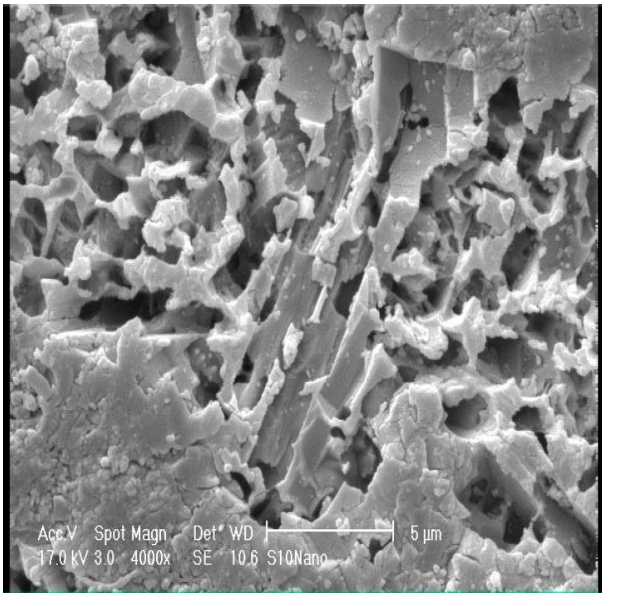

Fig. 7 SEM image of the size of cross sectional cavities in WA_{10} (×4000).

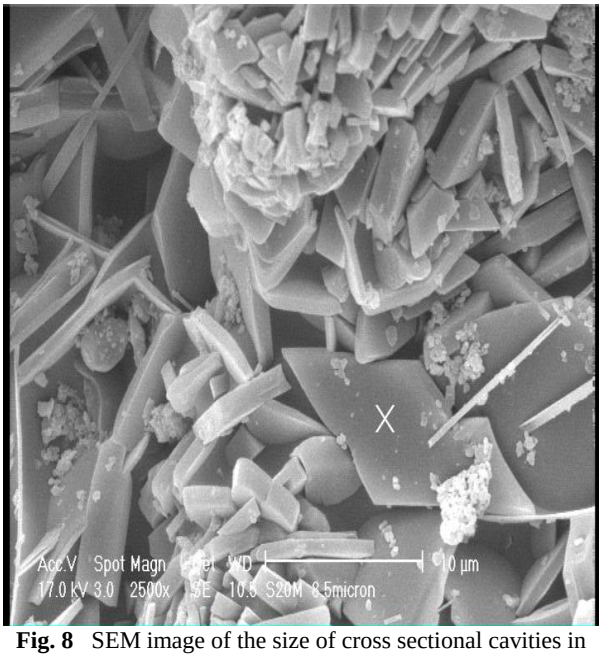

Fig. 8 SEM image of the size of cross sectional cavities in WA_{20} (×2500).

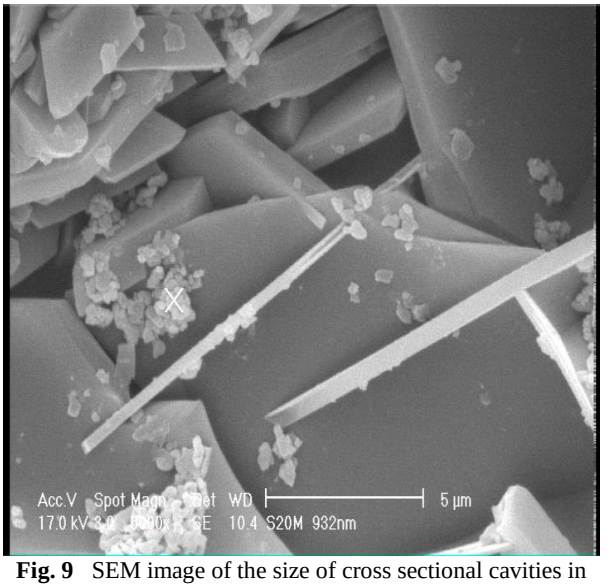

Fig. 9 SEM image of the size of cross sectional cavities in WA_{20} (×5000).

In short, it can be concluded from scanning electron microscope images:

- Diameter size of Wollastonite crystal particle and cavities in average and scattered was randomly measured by scanning electron microscope and measurement software from different particles of wollastonite crystals.
- In $WAn_{2.5}$ compositions, by adding nano-alumina as the second component, in comparison with base glass, it is concluded that the porosity has augmented.
- Trace amounts of Wollastonite crystal rod are also found.
- If the amount of the second component in all composites increases, sum of SiO_2+CaO will decrease and micron/Nano-sized alumina amount will increase.

According to the results of XRD ("Figs. 3 and 4"), Anorthite crystal plates owing to a combination of alumina and a small amount of wollastonite are seen. By using images of reversed Electron and phase analysis of bright and dark spots in the Figures of WA_{20} composition, it can be said that in terms of type, dark and bright spots are almost identical. SEM images are seen in "Figs. 5 to 9".

3.4. Mechanical Properties

Mechanical properties and relative density of sintered composites are summarized in "Table 3". As you can see, composites with second micron-sized alumina component have less relative density than wollastonite glass-ceramics. Also, second component size reduction has been effective in increasing the density and $WAn_{2.5}$ compositions which has the highest relative density of 94.13 percent, the highest flexural strength and fracture toughness and in comparison with wollastonite glass ceramic is optimized without the second component and has very good mechanical properties. Also, in the composites with second Nano-sized alumina because ferrite was 5, initial density would have better results in improving relative density. Comparing compositions of $WAn_{2.5}$ and $WA_{2.5}$, by reducing from 2 to 40 nm, mechanical properties of glass-ceramic composites have changed extraordinarily.

In the composites with second micron-sized alumina, high hardness is because of the high percentage of second hard phase. The low hardness of the rest of the composites than glass-ceramics is owing to the low relative density and increased porosity. In the composites with second Nano-sized alumina in $WAn_{2.5}$ and WAn_5,

Table 3 Mechanical properties and relative density of the samples

Samples	Vickers hardness (Gapa)	Flexural strength (MPa)	Fracture Toughness $\left(MPam^{\frac{1}{2}}\right)$	Relative density (%)
wollastonite glass–ceramic	6.68±7.98	120± 8.01	0.95 ± 0.07	95.37
$WA_{2/5}$	4.84 ± 1.14	50 ± 10.26	0.74 ± 0.08	88.28
WA_5	5.01 ± 1.66	81 ± 25.26	0.72 ± 0.14	83.27
WA_{10}	5.42 ± 1.62	88 ± 15.30	0.72 ± 0.22	79.56
WA_{20}	9.36 ± 1.15	45 ± 5.1	0.65 ± 0.35	74.52
$WAn_{2/5}$	7.92 ± 2.75	133 ± 20.07	1.40 ± 0.10	94.13
WAn_5	7.50 ± 8.05	114 ± 20.11	1.29 ± 0.14	91.63
WAn_{10}	6.65 ± 4.00	103 ± 15.20	1.21 ± 0.28	86.37

high hardness is owing to the high relative density and high toughness of the second component (α- Al_2O_3) than the glass-ceramics. In WAn_{10} low hardness is because of low porosity. The fracture strength of wollastonite glass-ceramic composites with second Nano-sized component is reduced by increasing weight percentage of second component. Also, the fracture strength of wollastonite glass-ceramic composites with second micron-sized component has increased by increasing weight percentage of second component and then has declined to 20wt%. By comparing all composites with wollastonite glass-ceramic, it can be concluded that only $WAn_{2.5}$ composite has higher flexural strength.

Strength of all nano-composites compared to micron composites is because of a higher density. Glass at sintering temperature of 750 °C has a bulk density of 2.76, a relative density of 98.43 and the flexural strength of 62.9. It is evident that in virtue of Crystallization, glass has a little strength. It can be concluded that if

sintering temperature of the powder samples was lower than crystallization temperature of the wollastonite glass, we would not have good mechanical properties [19-22].

In WA20, because of very small cracks that were visible with the naked eye, it had a very low flexural strength. The low hardness of micron composites than glass-ceramics is owing to the low relative density and increased porosity. This is the maximum reduction of 20%. High flexural strength of WA_{10}>WA_5>$WA_{2.5}$ is because of higher percentage of the second phase with a high elastic modulus. In $WAn_{2.5}$ it is owing to its higher relative density than the rest of composites. The low hardness of WAn_5 and WAn_{10} is because of the low relative density and increased porosity. As can be seen in all three, the percentage of nano-composite strength is four percent higher than micron composite.

Fracture toughness of wollastonite glass-ceramic composite with Nano-sized as well as micron-sized second component is reduced by increasing the weight percentage of the second component. By comparing all composites with wollastonite glass-ceramic, this conclusion can be drawn that only $WAn_{2.5}$ composite has higher flexural strength.

Improvement of mechanical properties of the $WAn_{2.5}$ composite can be on account of multiple mechanisms actions such as crack deflection and crack branching. The presence of tiny cracks in WA_{20} sample can be caused by differences between second component of the elastic modulus and context phase or difference between thermal expansion coefficients of the two components that have led to loss of mechanical properties.

4 CONCLUSION

The study has the following results:

1- The bulk density and relative density of all composites have been reduced. Comparing the relative density of the samples prepared from different powders showed that the reduction of particle size of second component from micron to Nano would boost the density and reduce porosity.

2- Adding 2.5 percent nano alumina improves flexural strength. By adding Al_2O_3 to Wollastonite and conducting heat treatment to a temperature of 1030°C alumina and wollastonite, phases are formed. Anorthite phase is formed at a temperature of 1090 °C and at 1170 °C, the main phase is crystalline.

3- Sintering temperature for samples prepared from different powders is above the crystallization temperature of the wollastonite glass-ceramics, which helps to improve mechanical properties of composites.

4- Shape, size and amount of the second component as well as shape and size of voids in wollastonite crystalline and consistent and complementary of these two with each other are important factors in enhancing the strength of composite and better optimization of mechanical properties of the composites and designing in general.

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