

FRACTOGRAPHY OF STIR CASTED Al-ZrO₂ COMPOSITES*

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Abstract– In this study, Al-ZrO₂ composites were produced by Vortex method using ZrO₂ powder with 1 micron average diameter as reinforce particles and Al-356 as the matrix metal. The melt composites were stirred for 13 minutes, then casted into a metallic mold. Different samples of 5, 10 and 15 volume percent of ZrO₂ in different casting temperatures of 750, 850 and 950°C were produced. The latter 2 casting temperatures are not a common practice but were chosen to enhance fluidity. Effects of volume percent of ZrO₂ particles and casting temperature on tensile strength, microstructure, and fracture surfaces of Al-ZrO₂ composites have been investigated. The highest tensile strength was achieved in the specimen containing 15 vol. % ZrO₂ produced at 750°C which shows an increase of 60% in comparison to the Al-356 non-reinforced alloy. Microscopic investigations of fracture surfaces revealed that fracture in a brittle manner with little or no necking happening. By increasing ZrO₂ content and casting temperature, the composites fracture goes in a more severely brittle manner.

Keywords– ZrO₂ particles, MMC, Vortex method, tensile strength, fracture surface

1. INTRODUCTION

Metal Matrix Composites (MMC's) are considered a group of advanced materials which represent low density, good tensile strength, high modulus of elasticity, low coefficient of thermal expansion, and good wear resistance. These characteristics could not be achieved together in the monolithic materials [1-3].

Vortex or stir casting is the most commonly used method to produce composite particulates. This is mostly due to its simplicity, low production cost and flexibility to produce a wide range of MMC's. Addition of hard ceramic particles into a ductile metallic matrix results in the production of composites that possess the properties of both phases [2-4].

Many researches have been done to study the effects of such second phases as SiC [5, 6], TiB₂ [7], Al₂O₃ [8], and B₄C [9] on reinforcing the aluminum matrix. All reports emphasize the positive effect of these materials on enhancing the mechanical properties of the resultant composites.

Zirconia is a refractory material with melting point of about 2680°C. ZrO₂ possesses good properties such as the low coefficient of thermal expansion, good thermal shock resistance, high melting point, low thermal conductivity, and excellent thermodynamic stability. Its density, Young's modulus, and hardness are 5.76 g/cm³, 190 GPa, and 1200 HV, respectively [10-14].

Production of Al- ZrO₂ composites by stir casting is associated with problems such as low wet ability of ZrO₂ by molten Al and a higher density of ZrO₂ compared to that of Al, which results in deposition and therefore non-uniform distribution of ZrO₂ particles. Based on the reasons mentioned above, at the time of

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this investigation, no successful production of Al-ZrO₂ composites via Vortex method has been reported. In the present study, Al-ZrO₂ composites were produced by optimizing the Vortex method parameters such as stirring duration. The effects of ZrO₂ content and casting temperature on the tensile strength of the composites were studied. Also, microscopic studies were carried out to study the fracture surfaces of the composites, which were not previously investigated.

2. EXPERIMENTAL PROCEDURES

The major raw materials used in this study were aluminum alloy and zirconia powder. Al-356 aluminum alloy (Kian Alloy Co.) was used as the matrix of the composites. The chemical composition of this alloy is shown in Table 1. Yttria stabilized zirconia powder (ZrO₂-3mol%Y₂O₃, D₅₀=0.79 μm, Tosoh Co.) was also used as reinforcing phase (Fig. 1).

Table 1. Chemical composition of Al-356 alloy

Element	Al	Si	Fe	Cu	Mg	Mn	Zn	Ti	Ni
Mass%	91.73	7.23	0.32	0.18	0.38	0.02	0.05	0.01	0.05

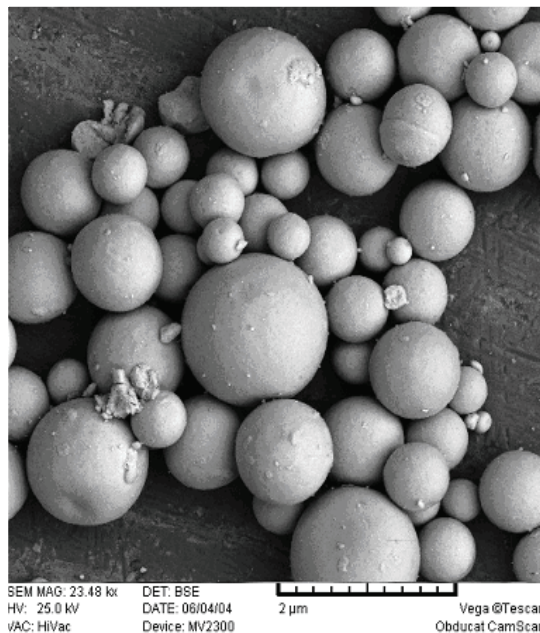


Fig. 1. Stabilized zirconia powder by Yttria (ZrO₂-3mol%Y₂O₃)

The furnace used in this research as shown in Fig. 2 and 3, included an impeller which was made of graphite. In order to produce the composites, the aluminum alloy was melted at 750, 850, and 950°C. The melt was stirred at a constant speed of 300 rpm for 13 min. The required amount of zirconia powder was weighed according to the weight of the treated melt (5, 10, and 15 vol. %) and then capsulated within a few aluminum foil wrappings and added into the molten alloy. Stirring was carried out for 2 more minutes and the resultant slurry was then cast into the metallic mold to solidify and form the composite specimens.

In order to investigate the tensile strength of the specimens, Instron 1195 tensile test was used. The tensile specimens were ground according to ASTM B557 (Fig. 4) [15]. For each testing condition, 5 specimens were subjected to tensile test and the average of the 5 results was reported.



Fig. 2. The casting system used in this study

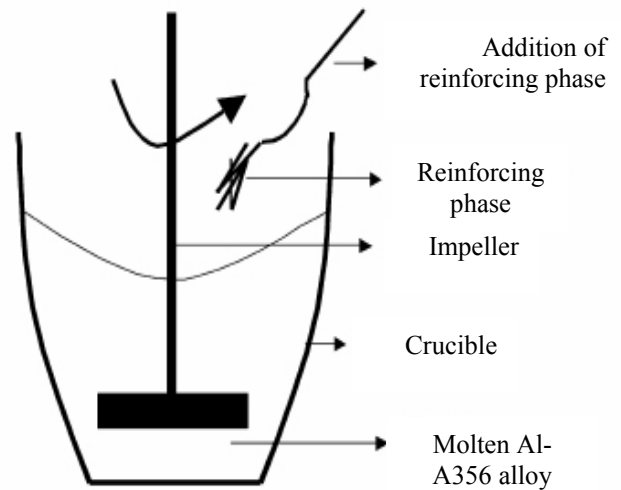


Fig. 3. The schematic of the Vortex method

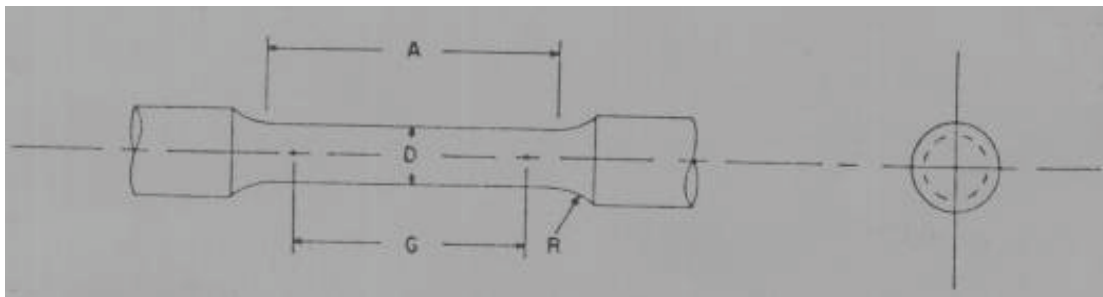


Fig. 4. The schematic of a tensile specimen (D=9 mm, G=45 mm, R=8 mm, A=54 mm) [15]

In order to investigate the fracture surfaces of the composite specimens, topographic observations were carried out using a CamScan MV2300 SEM. The fractured specimens of the tensile tests were cut into a disc of 10 mm thickness (Fig. 5).



Fig. 5. The specimens prepared for topographic observations

3. RESULTS AND DISCUSSIONS

a) Tensile test results

The effects of casting temperature and ZrO₂ content on the ultimate tensile strength (UTS) of the specimens are shown in Fig. 6. As it can be seen, at 750°C and 15 vol. % ZrO₂, the strength has increased to 232 MPa, which shows an increase of about 60% over the pure Al-356 alloy. The effects of the casting temperature and ZrO₂ content on the mechanical properties of the Al- ZrO₂ composites are shown in Table 2.

Table 2. Integrated results of mechanical properties of Al-ZrO₂ composites

Casting Temperature (C°)	Vol. % ZrO ₂	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Strain to Failure (%)
750	5	64	155	1.5
	10	80	188	2
	15	95	232	3.1
850	5	77	187	1.9
	10	77	187	1.9
	15	62	154	1.5
950	5	67	163	1.6
	10	74	180	2.2
	15	69	169	2

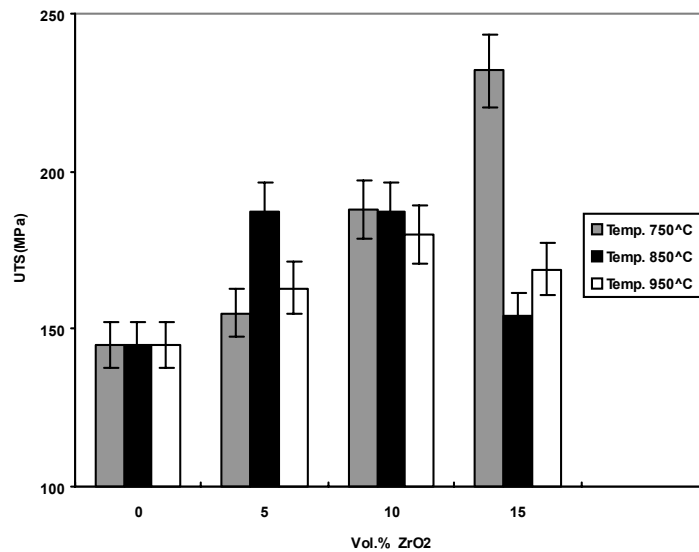


Fig. 6. The ultimate tensile strength of the composite specimens containing 5, 10 and 15 vol. % ZrO₂ produced at 750, 850 and 950°C

The reason for increasing the yield strength at 750°C for the samples of 0-15 vol. % ZrO₂ could be increasing the dislocations density and their pile-ups behind the ZrO₂ particles which act as obstacles in the movement of dislocations. The greater the amount of ZrO₂, the larger the number of dislocations formed and thus, a higher yield strength is achieved. This is suggested by the uniform distribution of ZrO₂ particles within the matrix observed in [16]. The SEM micrographs of the specimen containing 5, 10 and 15 vol. % ZrO₂ produced at 750°C are shown in Fig. 7-9.

On the other hand, the reason for increasing the tensile strength at 750°C for the samples of 0-15 vol. % ZrO₂ is the increasing dislocations density. This is due to the difference between the coefficient of the

thermal expansion of ZrO₂ and the matrix alloy. CTE of aluminum and zirconia are $24.10^{-6}K^{-1}$ and $10.10^{-6}K^{-1}$ respectively, causing a high stress in the microstructure during cooling of the composite.

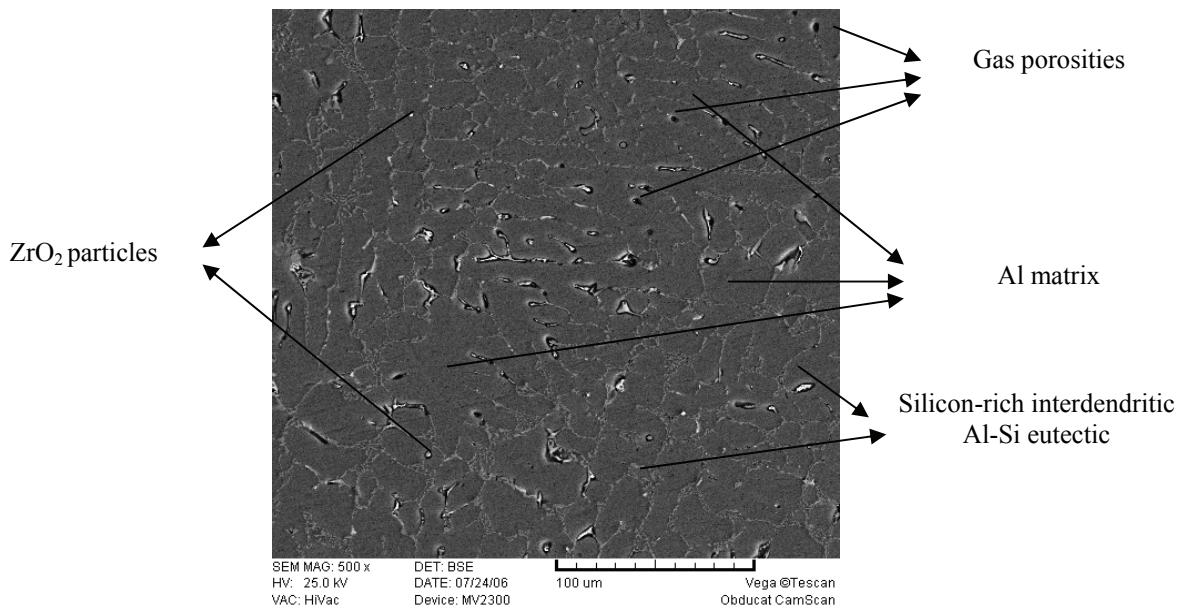


Fig. 7. The SEM micrograph of the specimen containing 5 vol. % ZrO₂ produced at 750°C

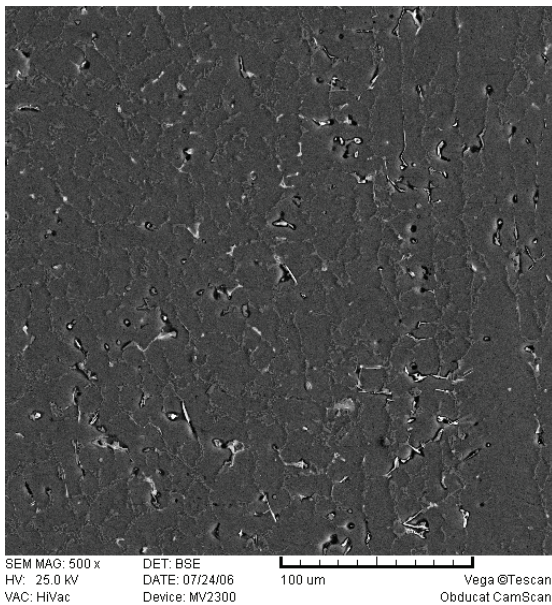


Fig. 8. The SEM micrograph of the specimen containing 10 vol. % ZrO₂ produced at 750°C

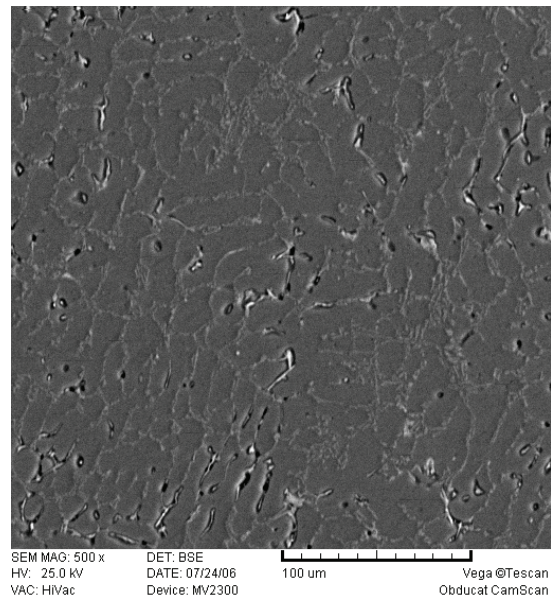


Fig. 9. The SEM micrograph of the specimen containing 15 vol. % ZrO₂ produced at 750°C

At 850°C and 5 vol. % ZrO₂, the tensile strength increases up to 30%, but almost levels off between 5-10 vol. % ZrO₂ and even slightly decreases at 15 vol. % ZrO₂. This is mainly due to the formation of porosities as the result of high temperature agitation and the increased amount of ZrO₂ particles [17]. The SEM micrographs of the specimens containing 5, 10 and 15 vol. % ZrO₂ produced at 850°C are shown in Fig. 10.

At 950°C, by increasing zirconia particles up to 10 vol. %, the tensile strength of the composite increases up to 30%, but it decreases when the volume percent of zirconia particles increases from 10 to

15 vol. %. This happens because of the porosities that are created by such temperature via favoring the fluidity and agitation of the melt. Also, increasing the volume percent of zirconia enhances the viscosity of the melt which causes the gas to be trapped into the melt and the porosity of the composite to be increased. This results in the strength of the composite to be decreased. Nevertheless, the porosity in the stir casting process is expected. The SEM micrographs of the specimens containing 5, 10 and 15 vol. % ZrO_2 produced at 950°C are shown in Fig. 11.

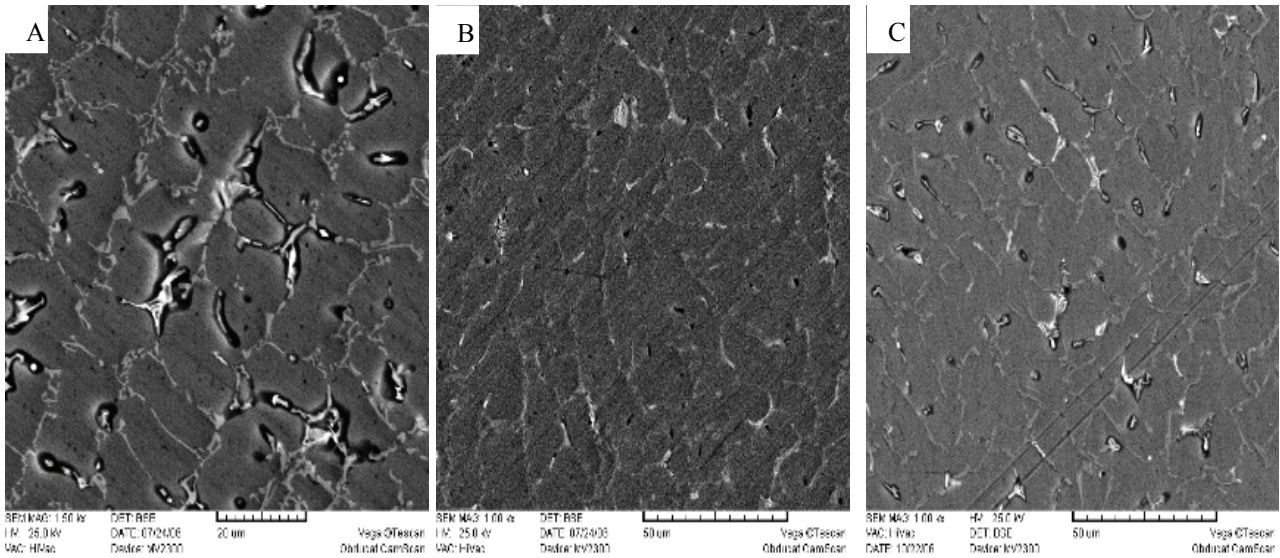


Fig. 10. The SEM micrographs of the specimens produced at 850°C containing A) 5, B) 10 and C) 15vol. % ZrO_2

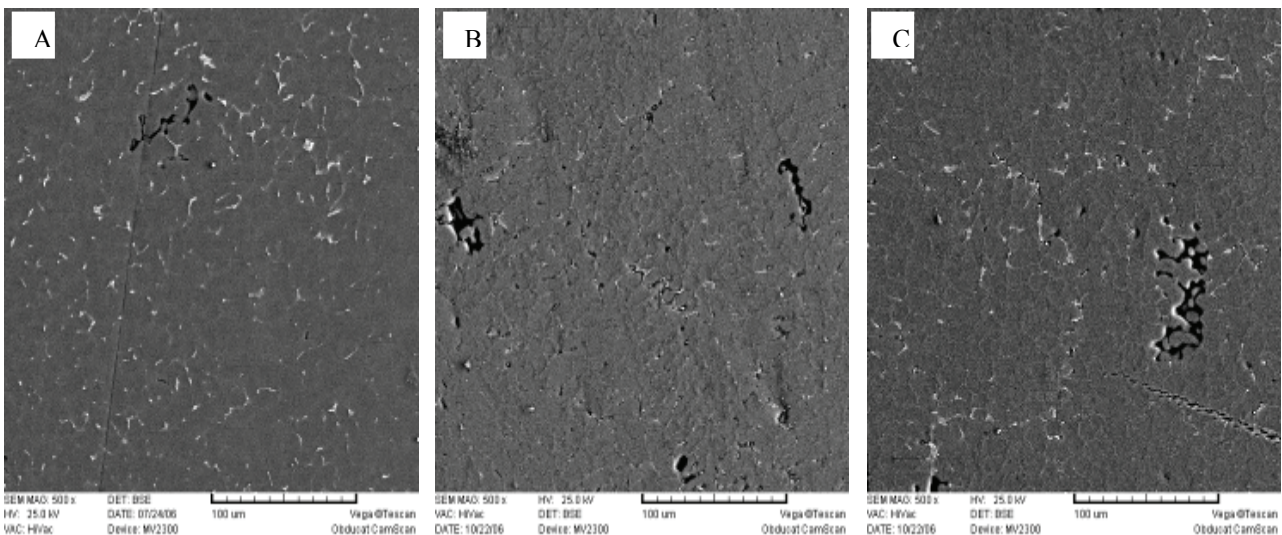


Fig. 11. The SEM micrographs of the specimens produced at 950°C containing A) 5, B) 10 and C) 15vol. % ZrO_2

The distribution pattern of the reinforcing particles and also their wettability with molten alloy has the greatest significance [18, 19]. Figure 6 shows that decreasing the strength of the samples with 15 vol. % of zirconia at 950°C compared to the samples with 10 vol. % is less than the samples prepared at 850°C. This could be related to greater wettability of zirconia at 950°C, thus zirconia is more incorporated with 15 vol. % at 950°C compared to 850°C.

At high temperatures, the wettability is enhanced (since the molten alloy possesses higher fluidity), but porosities and segregation are also more likely to form since the agitation of the melt is higher. This

can also affect the distribution of the particles, resulting in less entrapment of the particles and less of the remaining zirconia into the melt composite. Also, undesirable chemical reactions might take place at elevated temperatures [20, 21]. The temperature of the melt is effective on the final microstructure and morphology of the phases; with a higher temperature the probability of formation of a dendrite structure and so the related segregation increases. This is effective on the mechanical properties. Therefore, the effects of these factors should be considered altogether in order to study the tensile strength changes in each casting condition.

It seems that the optimum condition for production of Al-ZrO₂ composites is 750°C and 15 vol. % ZrO₂ under which a uniform distribution of ZrO₂ particles and also the maximum tensile strength is achieved.

b) Fracture surface

Figure 12-14 show the SEM micrographs of the fracture surfaces of the specimens containing 5, 10 and 15 vol. % ZrO₂ produced at 750, 850 and 950°C. These images are taken by secondary electrons in order to study the fracture mechanisms of Al- ZrO₂ composites.

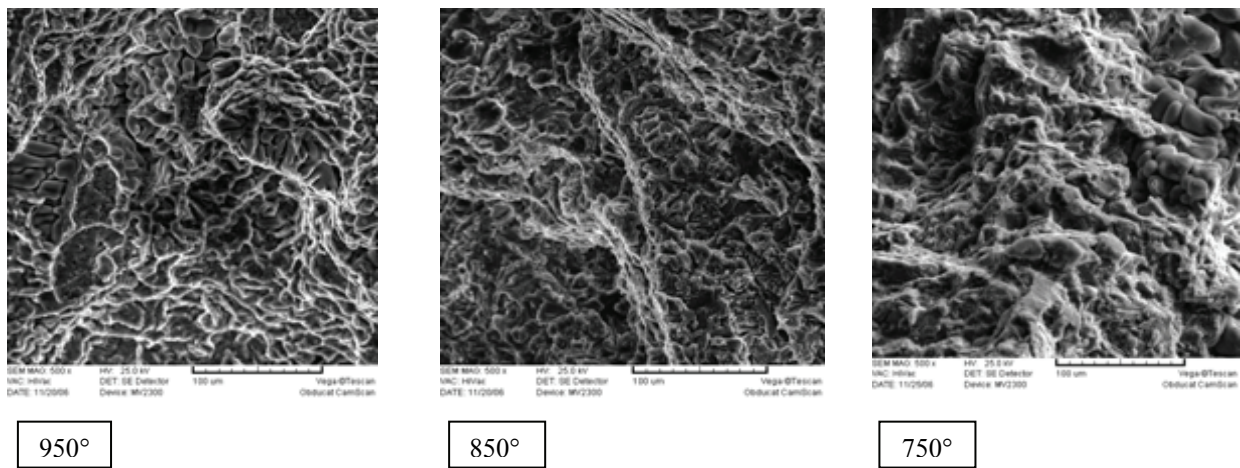


Fig. 12. Topographic images of the fracture surfaces of the specimens containing 5 vol. % ZrO₂ produced at different temperatures

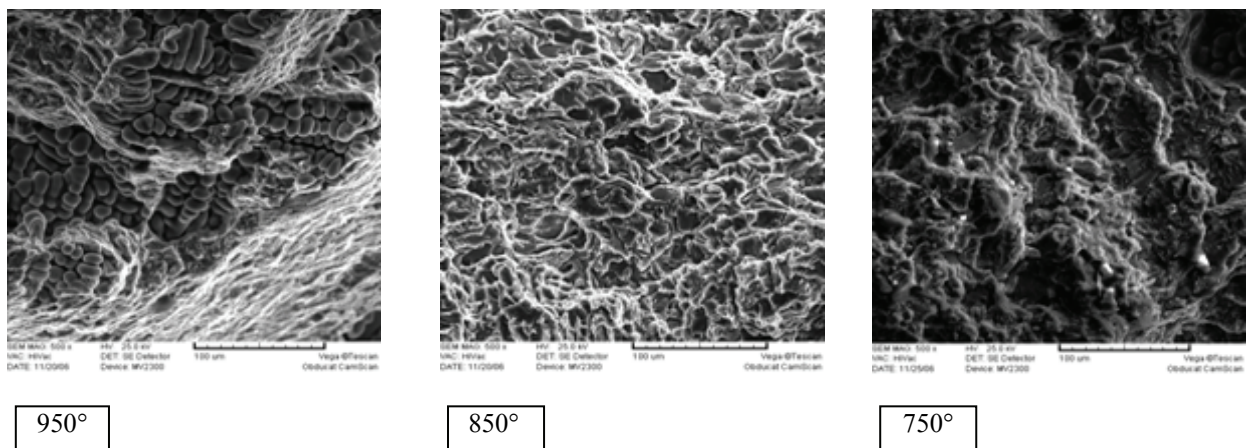


Fig. 13. Topographic images of the fracture surfaces of the specimens containing 10 vol. % ZrO₂ produced at different temperatures

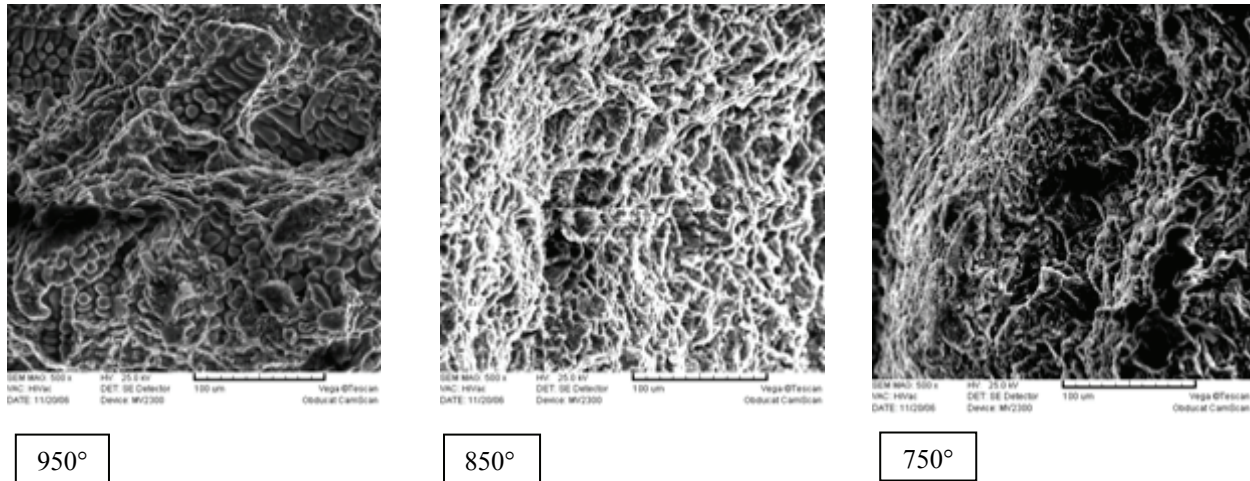


Fig. 14. Topographic images of the fracture surfaces of the specimens containing 15 vol. % ZrO_2 produced at different temperatures

Fracture usually occurs as the result of one or a combination of the following mechanisms.

- 1) Fracture of the reinforcing particles [22];
- 2) Partial debonding of particle-matrix interface and nucleation of voids [23];
- 3) Growth of the voids and initiation of cracks in the matrix [24].

The fracture observed in the composites depends on a variety of factors including: the processing method, post heat treatments, the applied stress, distribution and morphology of the reinforcing particles, etc.

In brittle fracture, reinforcing particles are not observed in surfaces, while eutectic phases are mostly present. Crack initiation takes place from these areas and the fracture surfaces of the composite contains almost no fractured ZrO_2 particles, but instead, display evidence of the eutectic and particle pullout (Fig. 12-14). These eutectic phases appear brighter in the SEM images than other areas. But in ductile fracture, the reinforcing particles start to crack first and then crack growth takes place from these particles. This is the reason for observing fractured pieces of reinforcing particles in topographic images [25-28].

According to the SEM images and existence of the eutectic Al-Si phase (Silicon-rich interdendritic), fracture of Al- ZrO_2 composites is brittle and the third mechanism is dominant. During solidification, the ZrO_2 particles are rejected to the solid-liquid interface and therefore are trapped and clustered in the interdendritic Al-Si eutectic. Accumulation of ZrO_2 particles and Al-Si eutectic (which are rich in Si) are locations in which crack initiates. Fracture of Al- ZrO_2 composites initiates from the matrix. In other words, fracture of the composite is controlled by fracture of the matrix [29, 30].

Based on the above observations and explanations, the fracture of Al- ZrO_2 is brittle, with little or no necking observed around the fracture site and by increasing the casting temperature and ZrO_2 content, the fracture becomes even more brittle. This is due to the increased amount of bright Si rich areas observed in the SEM images [25, 31-33].

4. CONCLUSION

- 1) Al- ZrO_2 composites were successfully produced by stir casting in this study.

- 2) By increasing the ZrO₂ content in the specimens produced at 750°C, the tensile strength also increases. This is due to the accumulation of dislocation behind ZrO₂ particles which act as barriers on the movement of dislocations.
- 3) Increasing the ZrO₂ content in the specimens produced at 850 and 950°C has almost no significant effect on the tensile strength.
- 4) According to the topographic observations, the fractures of Al-ZrO₂ composites are brittle, since the matrix fracture is dominant and almost no fractured ZrO₂ particles are observed.
- 5) The optimum production conditions of Al- ZrO₂ composites are 750°C and 15 vol. % ZrO₂.

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