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# Microstructure and Mechanical Properties of Aluminum Alloy Matrix Composite Reinforced with $ZrO_2$ Particles

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Al-$ZrO_2$ composites have found more applications in aerospace and automotive industries due to their higher tension and wear properties. One of the demanded methods in manufacturing these composites is vortex casting, because it is less expensive and covers large variety of materials and fabricating situations. In this research, a casting system was designed for manufacturing Al-$ZrO_2$ composites in 5, 10 and 15 vol. % $ZrO_2$ and 750, 850 and 950 °C casting temperatures. The resulting composites were tested for their mechanical properties and microstructure to study the effect of $ZrO_2$ and temperature parameters. The results show enhanced mechanical properties. In this case the best sample (UTS = 232 MPa, H = 64 HB) was reached at 750 ºC and 15 % $ZrO_2$ (from 145 Mpa and 43 HB, respectively). Generally it can be said that increasing $ZrO_2$ vol. % and elevating melt temperature result in higher percent of porosities, uniform distribution of them and also increased segregation. By increasing $ZrO_2$ % and shortening casting time, an enhanced distribution of $ZrO_2$ particles can be achieved.

**Key Words: Vortex casting, Ceramic particles, Al-$ZrO_2$ composites, Aluminum, Zirconia, Properties.**

## INTRODUCTION

Generally, composite materials are divided into three major categories *viz.*, metal matrix composites, polymer matrix composites and ceramic matrix composites[1].

Metal matrix composites are considered as a group of advanced materials which represent low weight, high strength, high modulus of elasticity, low coefficient of thermal expansion and good wear resistance. These characteristics could not be achieved together in monolithic materials[2].

Stir-casting technique for producing metal matrix composites (MMCs) has been developed to manufacture a wide range of engineering components due to its simplicity, economy and flexibility[3]. While this technique is accompanied by some difficulties such as processing parameters control, undesired chemical reactions in the boundary of matrix metal and ceramic particles and low wetability of ceramics by metal[4].

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Among the ceramics, zirconia isn't exception. Its low wetability by aluminum produces the difficulties in fabricating of $Al/ZrO_2$ composites. Zirconia is a refractory material with a melting point about 2680 ºC[5]. This material has good properties such as low coefficient of thermal expansion, good resistance against thermal shocks, high melting point, low thermal conductivity, excellent thermodynamically stability and high radiation[6,7]. But zirconia has the allotropic deformation in different temperatures. Its crystallographic structure is monoclinic[8] till 1170 ºC.

The solidification microstructure of the composite is important because it establishes the distribution of reinforcing particulates. Subsequent fabrication processes may modify the distribution, but the more uniform is the initial as-cast billet, the more homogeneous will be the final composite[9]. The behaviour of particulates at the solid/liquid interface has attracted the interest of many researchers over the past several years. This is because the mechanical properties of particulate composites are mainly controlled by the distribution of the particulates. Therefore, the most important characteristics are due to interaction of particulates with the solid/liquid interface. A uniform distribution is required for the strengthening of the composites[10]. The distribution of particulates in MMCs manufactured by casting technique depends to a great extent, on the nature of interaction between ceramic particulates and the rowing solidification front. Basically, when a moving solidification front intercepts on insoluble particulate, it can either push it or engulf it[11].

The influence exerted by the matrix microstructure on the mechanical properties of MMCs has been emphasized with experimental evidence[12]. This in turn highlights the practical importance of solidification, in governing the final composite microstructure. Direct transposition of rules developed for microstructural control in the solidification of unreinforced metals is not possible with MMCs because the reinforcing phase frequently modifies solidification of the matrix.

Thus, the formation of solidification microstructure in cast particulate composites is mainly influenced by nucleation or its absence, on particulates and particulate pushing or engulfment by the solidification front.

In spite of the extensive research done world-wide over the last quarter of century on cast MMCs, understanding of the phenomena occurring during solidification of these advanced materials is far from complete.

In the present study, by 9 distinct and different casting conditions, a variety of samples were produced to be used for density, tensile and hardness tests and also SEM and XRD analysis. Aluminum A356 and zirconia micron particles were selected as raw materials.

## EXPERIMENTAL

The major raw materials used in this research were aluminum and zirconia with the characteristics as below: Aluminum (Al-356) was prepared by Kian Alloy Company-Kashan-Iran as metal matrix of composites. The chemical composition of this alloy is shown in Table-1. Yttria stabilized zirconia powder ($ZrO_2$-3 mol % $Y_2O_3$, $D_{50}$ = 0.79 μm): prepared by Tosoh Company-Japan as reinforcing material.

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 |

Aluminum was melted inside a crucible in electric furnace at 750, 850 and 950 ºC. The melt was stirred with a graphite stirrer at a constant rotation speed of 300 rpm. Stirring time was 13 min. About 3 g cryolite was plunged into the melt to improve the foundry condition and prevent from slag formation. Stirring was continued for a few minutes. The 5, 10 and 15 vol. % zirconia was added while the melt was stirred. Molten composite was poured inside a metallic mold. This kind of mold was used to prevent undesired conditions and to increase the solidification speed because of the metallic structure and samples size (cylindrical shape with 15 cm height and 15 mm diameter).

In order to investigate the present phases in the samples, XRD (PW-1800 model, Philips) was used. The microstructures were determined by scanning electron microscopy (Camscan-MV2300 Model, Oxford). Specimens were polished and etched using keller solution[13].

In order to measure of the density of samples by Archimedes method, a density test system (Sartorius model) has been used.

The hard meter system DVRB-M model made by Eseway Company, England has been used to determine Brinell hardness of samples. After grinding samples, they were polished up to 1 µ. Tests were made with the load of 31.25 Kgf and the punch diameter was 2.5 mm. In order to determine hardness value, two samples were selected of each casting and each sample was tested 5 times. The average of results, determined the hardness value.

In order to measure tensile strength of samples, the tensile test system 1195 model made by Instron Company, England has been used. Tensile tests were done in order to study the tensile strength of samples. Samples were made in a cylindrical shape based on ASTM.B557 standard[14].

## RESULTS AND DISCUSSION

**XRD Tests:** Present phases in the sample produced at 850 ºC-10 vol. % are shown in Fig. 1. It can be seen that zirconia and aluminum are present in this sample and the other phases which might be present because of destructive chemical reactions, are absent. Because in the XRD results of the other samples exactly the same patterns exist, with the difference that the pick of patterns depends on the volume percentage of zirconia, only the result of above sample is reported as an example.

**SEM Tests:** The SEM micrographs and X-ray maps of the samples produced at 750, 850 and 950 ºC are shown in Fig. (2-10), respectively. In these figures the black matrix is aluminum and the white spots represent $ZrO_2$ particles. The presence of silicon laminate in the aluminum matrix can clearly be observed (white plate) in all samples. The existence of the agglomerates in the composites is confirmed by the figures.

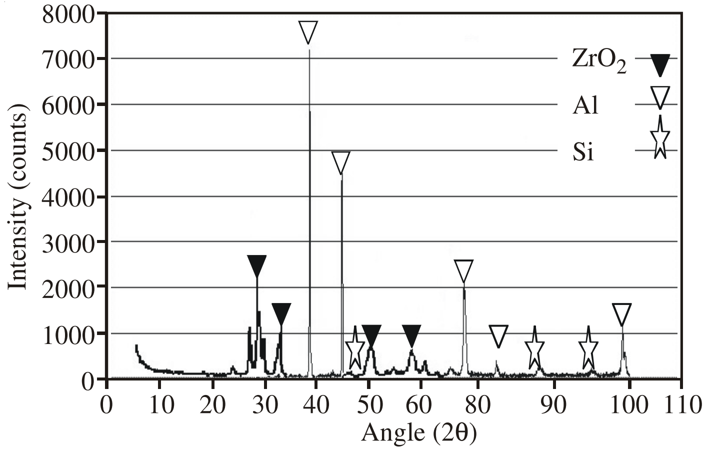

Fig. 1. XRD patterns of (Al-ZrO$_2$) composite casted at 850 ºC-10 vol. %

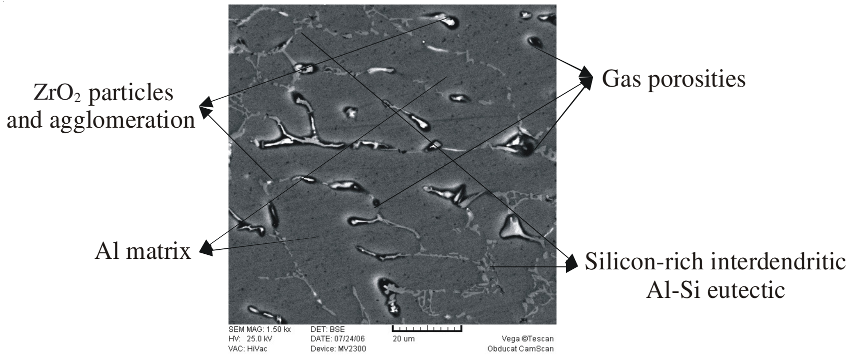

Fig. 2. SEM micrograph of (Al-ZrO$_2$) composite casted at 750 ºC-5 vol. %

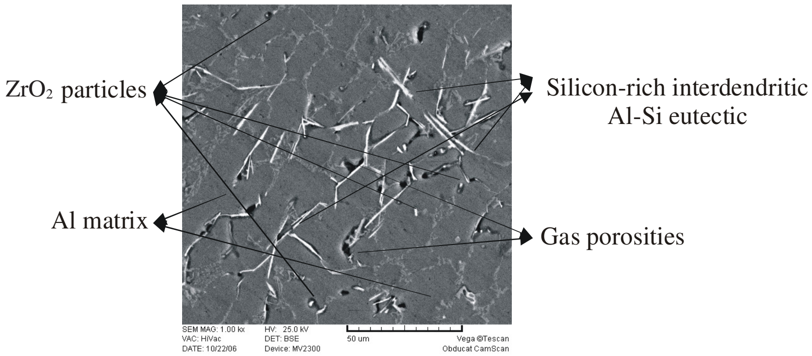

Fig. 3. SEM micrograph of (Al-ZrO$_2$) composite casted at 750 ºC-10 vol. %

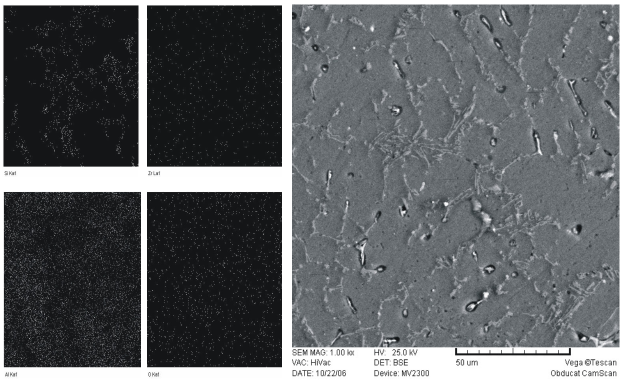

Fig. 4. SEM micrograph of (Al-$ZrO_2$) composite casted at 750 ºC-15 vol. % with X-ray map

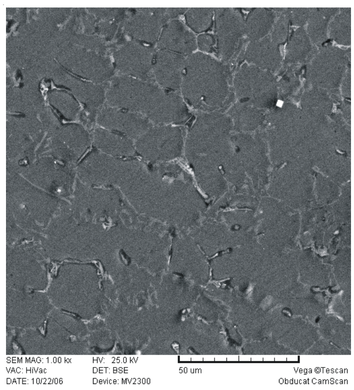

Fig. 5. SEM micrograph of (Al-$ZrO_2$) composite casted at 850 ºC-5 vol. %

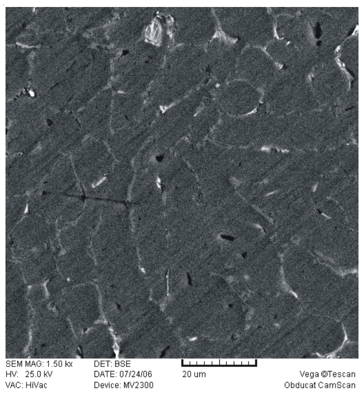

Fig. 6. SEM micrograph of (Al-$ZrO_2$) composite casted at 850 ºC-10 vol. %

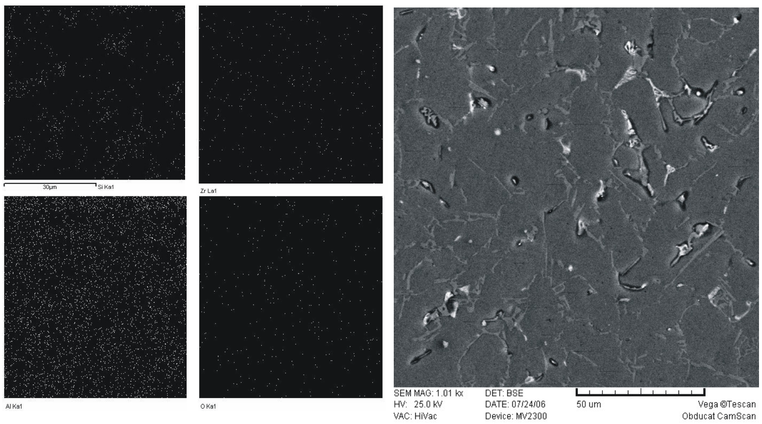

Fig. 7. SEM micrograph of (Al-$ZrO_2$) composite casted at 850 ºC-15 vol. % with X-ray map

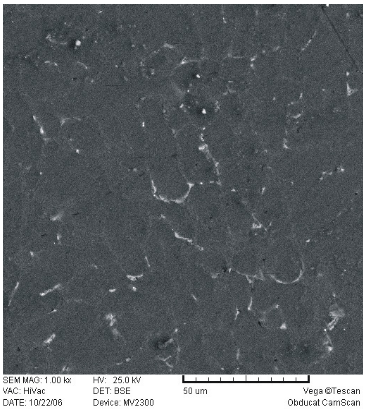

Fig. 8. SEM micrograph of (Al-$ZrO_2$) composite casted at 950 ºC-5 vol. %

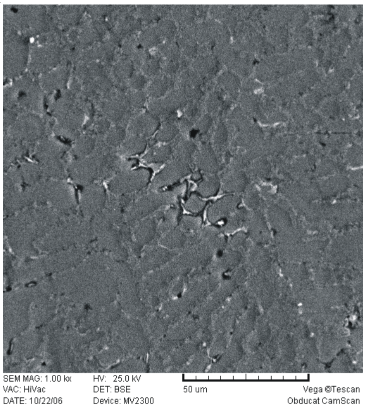

Fig. 9. SEM micrograph of (Al-$ZrO_2$) composite casted at 950 ºC-10 vol. %

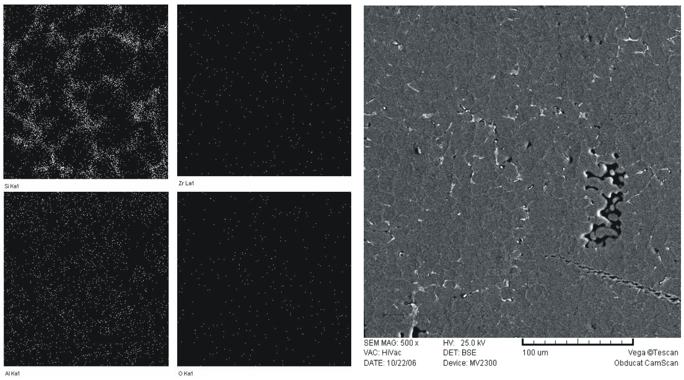

Fig. 10. SEM micrograph of (Al-$ZrO_2$) composite casted at 950 ºC-15 vol. % with X-ray map

This is a common problem in the composites manufactured by vertex method. This deficiency is worsening by particle size reduction. Despite the presence of the relatively small agglomerates, they are dispersed uniformly across the composites. The distribution of Zr and O (represent of $ZrO_2$ particles) can be observed in the X-ray maps.

The interface between solid and liquid phases moves during solidification. In this step, particles may be entrapped in the solid phase or pushed away by the surface of the solid phase[15]. In a rare case, nucleation may occur on the surface of $ZrO_2$ particles which is identified as engulfment mechanism. The main requisite for this mechanism is appropriate wetability between particles and matrix. Due to the inadequate wetability between $ZrO_2$ particles and Al matrix, engulfment mechanism can not be taken place. Therefore, a combination of first (entrapment) and (mainly) second (push away) mechanisms is dominant in this system.

Surface of metallic mold is a suitable position for heterogeneous nucleation, because they are rapidly cooled during solidification[16]. Therefore, surfaces of the $ZrO_2$ particles are not nucleation place and as a result engulfment mechanism can not be occurred. During solidification, alloying elements (mainly Si) and reinforcement powder ($ZrO_2$ particles) are pushed away by liquid/solid interface. Due to this phenomenon Si crystals are transferred to the surface of agglomerates ($ZrO_2$ particles) and are solidified at those places. Nucleation of Si on the surface of $ZrO_2$ particles can be observed in all specimens that in some area both of them overlap each other in SEM micrographs. Moreover, continues growth of eutectic phase is prevented by $ZrO_2$ particles[15].

The superiority of entrapment mechanism is enhanced with $ZrO_2$ amount increasing, while the supremacy of push away mechanism is reduced with additive level increasing. Therefore, as a result of additive rising, the amount of pushed away particles was decreased. Consequently, $ZrO_2$ particles were entrapped between the dendrites arms which led to more uniform particle distribution across the composite[17].

Processing temperature, volume fraction of additive, solidification time, mixing rate, mixing time and particle size are the most important factors on homogeneity of particle distribution in the matrix. Uniform particle distribution of $ZrO_2$ particles in the present study is probably due to water quenching and high thermal conductivity of the steel mold which causes rapid solidification and short distance between dendrite arms[15].

As it can be seen in the figures, all samples contain pore which is formed because of small size of the $ZrO_2$ particles. Surface area of the particles is increased with particle size reduction which causes viscosity rising, air entrapment and pore's volume fraction increasing[18,19]. The volume fraction of pores also increased with additive amount ($ZrO_2$ powder) increasing, which is because of the particle agglomeration. In addition, increasing the volume fraction of additive increases viscosity of the melt and as a result the pore's volume fraction[20,21]. Furthermore, the pore formation in vortex method is unavoidable and the amount of pore's volume fraction in the composites produced by this technique is usually high. Processing temperature and mixing time are other effective parameter on the pores' volume fraction. Increasing the processing temperature increases the wetability of the melt, as well as the destructive chemical reactions between Al matrix and $ZrO_2$ powder. Fortunately, there wasn't any destructive chemical reaction in this study. It also increases the air entrapment in samples, which enhances the amount of pores' volume fraction[22,23]. This phenomenon can clearly be observed in the figures.

Destructive effects of high processing temperature and high $ZrO_2$ volume fraction can be seen in sample produced at 950 ºC with 15 vol. % of $ZrO_2$ additive. It can be revealed from the results that the lowest mechanical properties are obtained in this specimen. The highest pore's volume fraction are measured in this composite (Fig. 10). The sample containing 15 vol. % of $ZrO_2$ additive, produced at 750 ºC demonstrates most appropriate properties, however when it was produced at 950 ºC demonstrates the lowest properties.

**Density tests:** According to densitometry graph in Fig. 11, in general, the temperature of 750 ºC is optimum, because of this fact that in this situation, the density increases with increasing of volume per cent of zirconia particles. This result is in according to the Mixture rule that means the presence of zirconia particles into the composite and also, increasing of total density with increasing of zirconia particles volume per cent. According to Mixture rule, the total density increases with increasing of volume per cent of second phase[24].

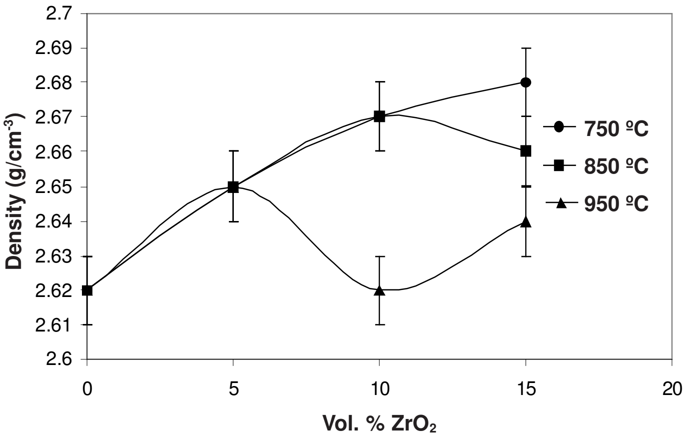

Fig. 11. Diagram of densitometry of cast samples at 750, 850 and 950 ºC containing 5 up to 15 vol. % reinforcing zirconia

Increasing the temperature from 750 ºC, a new factor (high temperature) for increasing the porosity of system, as well as increasing of volume per cent of ceramic particles, is added. At 850 ºC, there is a situation that shows an increasing and then a decreasing of density with increasing of volume per cent of zirconia particles. At this temperature, up to 10 volume per cent of reinforcing phase, the density increases, but it decreases with further increasing of zirconia particles that shows the destructive influence of high volume per cent of zirconia particles on increasing of porosity.

At 950 ºC, it seems to be a contradiction between temperature and volume per cent of ceramic particles (this phenomenon existes a little at 850 ºC). At this case, the effect of temperature compare to effect of volume per cent of particles is predominant and causes the contradictory behaviour of composite compare to 850 ºC case (the graph has a minimal point). Decreasing of density in this case means increasing of porosity because of high temperature which causes the high fluidity and turbulence of melt. At the most volume per cent of this temperature (15 vol. %), because of more viscosity of melt, the volume of gas entrapment is increased which increases the porosity and decreases the density. In this case, the effect of volume per cent of particles compare to effect of temperature is predominant and causes the increasing of density.

In general, during production of the composites by this stir casting method, a series of favourable and non-favourable factors exist which depending on predominant situations, cause the increasing or decreasing of properties and these changes are shown with increasing or decreasing of graphs. This is obvious in next graphs and results. The most important aim of this study, has been to optimize these factors to enhance the best properties, because these factors are dependent each other.

**Hardness tests:** The effects of temperature and additive amount on the hardness are shown in Fig. 12. It demonstrates that the hardness of Al-$ZrO_2$ composites is higher than that of the aluminum alloy (43 HB). This is because of extremely high hardness of $ZrO_2$ compare to the hardness of Al. Fig. 12 also illustrates that the hardness of the composites are decreased with temperature increasing. The hardness reduction rate is increased with the additive volume fraction increasing. Although it is expected that high temperature improves particle wetability and consequently composite's homogeneity and hardness, but other parameters such as pore formation, interface weakening, particle agglomeration and defect formation seems to be predominant at high temperature. Therefore, the hardness of the composites is reduced with temperature and volume fraction increasing.

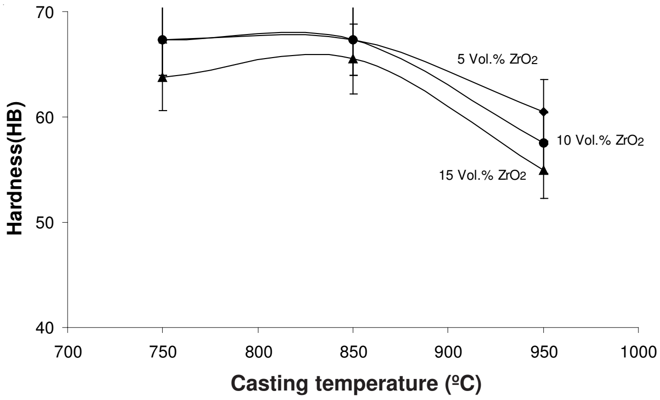

Fig. 12. Diagram of results of hardness test *versus* temperature and volume fraction of $ZrO_2$

**Tensile strength tests:** The effect of casting temperature and amount of $ZrO_2$ additive on ultimate tensile strength (UTS) of the samples are shown in Fig. 13. It can be seen that at 750 ºC with 15 vol. % of $ZrO_2$, the amount of ultimate tensile strength increased to 232 MPa which is *ca.* 60 % higher than that of the unreinforced Al (145 MPa).

Zirconia has monoclinic structure, while aluminum crystallizes in FCC structure. As a result of dissimilar crystalline structure of zirconia and aluminum, their interface seems to be incoherent[25,26]. Therefore, this incoherency increases energy of the system which may lead to stress generation and as a result strength enhancement. Presumably high strength of the Al matrix composites is due to the high work hardening rate of the composites during their strain. Work hardening enhancement can be related to the elastic properties of the $ZrO_2$ particles and their prevention from matrix plastic deformation. Therefore, in the presence of a strong interface, the $ZrO_2$ particles prevent matrix plastic deformation and consequently increase work hardening. Moreover, different thermal expansion coefficient of zirconia ($10 \times 10^{-6}$ $K^{-1}$) and aluminum ($16 \times 10^{-6}$ $K^{-1}$) creates stress which can increase dislocations density and as a result composite's strength. Also increasing of dislocations density and their pile-ups behind $ZrO_2$ particles act as obstacles in the movement of dislocations. The more the amount of $ZrO_2$, the more the number of dislocations formed and thus higher tensile strength is achieved[25,26].

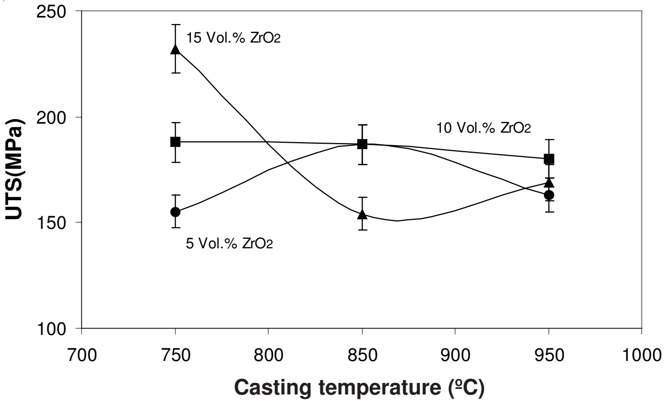

Fig. 13. Diagram of maximum tensile strength of cast samples at 750, 850 and 950 ºC containing 5 up to 15 vol. % reinforcing zirconia

Fig. 13 shows also the effect of casting temperature on tensile strength. It illustrates that in the samples containing 5 vol. % of zirconia, the tensile strength increases with temperature increasing up to 850 ºC. Conversely, above 850 ºC, the tensile strength decreases with temperature increasing. The tensile strength reduction might be due to generation of defect such as pores and particle agglomerates, which are produced at high temperature. In the composites containing 10 vol. % of zirconia, because of the same reason, the tensile strength decreases slightly with temperature increasing[27] from 750-950 ºC.

In the specimens containing 15 vol. % of zirconia, the maximum tensile strength was obtained at 750 ºC. However, with temperature increasing above 750 ºC the tensile strength decreased sharply. At temperature higher than 750 ºC and the highest volume fraction of $ZrO_2$ (15 vol. %), pore generation as a main reason reduces tensile strength. Segregation, interfacial separation, microcrack generation in the matrix and weakening by all mentioned defects at high temperature can also be considered as the other source of tensile strength reduction. Even in the worse condition the tensile strength of the reinforced Al is higher than that of unreinforced Al.

## Conclusion

Aluminum alloy was successfully reinforced with $ZrO_2$ powder having the grain size of 1 μm and Al- $ZrO_2$ composites were produced using vortex method. Water quenching, high thermal conductivity of the steel mold and small size of casted samples led to rapid solidification that results to uniform particle distribution. A combination of particle entrapment and particle (mainly) push away mechanisms is occurred during solidification. Extremely high hardness of $ZrO_2$ increases the hardness of Al-$ZrO_2$ composites. The tensile strength of Al-$ZrO_2$ composite was increased compared with unreinforced Al matrix. Because of the pore formation as a main reason, the hardness and the tensile strength of the composites were decreased with temperature and volume fraction of $ZrO_2$ increase.

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