Synthesis and characterization of zirconium dioxide particulate reinforced aluminium alloy metal matrix composite

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Aluminium alloys based metal matrix composites are evolving in industrial applications where high strength to weight ratio is required. In this research, 6061 grade aluminium alloy matrix with zirconium dioxide, particulate reinforced composite is fabricated. Composite material which reinforced with zirconium dioxide is fabricated with various weight percentages like 2%, 4%, 6%, 8% and 10% of reinforcement respectively, using stir casting process. Metallurgical and mechanical properties of the composite are analysed. Scanning electron micrograph showing that the particulates are dispersed uniformly into the matrix alloy. Particulate agglomeration is significantly reduced in the fabricated material. Addition of ceramic particulates improves the hardness of material by restricting dislocation of alloy matrix. Tensile test results show that the addition of zirconium dioxide, particulate increases its strength up to 6% addition of ZrO₂. Futher additon of zirconium dioxide, decrease its stength. The ultimate strength (UTS) of the AA60611 6% ZrO₂ composite were 169 MPa which is 24.26% higher than that of AA6061 alloy. The Microhardness of the AA6061 / ZrO₂ is found to be 32.73% higher than that of AA6061 alloy. Dry sliding wear behavior of AA6061/0-10 wt% ZrO₂ composite is investigated at room temperature by using a pin-on-disc wear testing apparatus. The possible sliding wear mechanisms were examined with the help of SEM micrographs of worn surface. When the wt% of ZrO₂ reinforcement in the matrix is maximized, wear mechanism of composite is found to be abrasive.

Keywords: Stir casting method, SEM, Tensile strength, Hardness, Wear rate, Wear SEM analysis

Metal matrix composites are evolving in industry for its properties like high strength, light weight, inexpensive processing, etc. Aluminium alloys are widely used in aerospace and automotive applications because of their high strength to weight ratio and corrosion resistance behavior. The metal matrix composites (MMC) of aluminium alloys improve the behavior of material by incorporating compatible reinforcements. Commercially available 6061 grade aluminium alloy as given in Table 1 is one of the widely used general purpose materials. It is preferred in wide applications because it has good mechanical and corrosion resistance behavior. This precipitate hardening aluminium alloy exhibits good weldability.

Many researchers have investigated aluminium alloy based composite materials fabricated using stir casting process¹. The objective of the work is to produce AA6061/TiC AMCs using stir casting and develop an empirical relationship incorporating the stir casting variables to predict the tensile strength intermediate range of parameters yielded castings with a homogeneous distribution of TiC particles and minimum porosity. The UTS were high when the porosity was low, and the distribution was homogenous.

Radha and Vijakumar² investigated the incorporation of SiC and graphite particles into a conventional aluminium alloy has shown results with increased elastic modulus, strength and elevated temperature capability relative to the baseline matrix. It increases with increase in reinforced particulate from 0% to 0.7% of graphite. The improvement in the hardness of the composites with increased content of reinforcement mainly attributed to the high hardness.

Suresh *et al.*³ investigated the various Al6061 composites prepared by reinforcing different weight fractions of TiB_2 and graphite and their mechanical and thermal properties of hybrid composite were also investigated. Their work is based on tensile testing using AE and also emphasizes on hardness, fatigue and thermal behavior of Al6061–TiB₂–graphite hybrid

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	Table 1 — Composition of ASTM AA6061 aluminium alloy													
Si	Fe	Cu	Mn	Mg	Zn	Cr	Ni	Ti	V	Pb	Zr	В	Al	
0.483	0.648	0.082	0.077	0.687	0.211	0.013	0.024	0.028	0.007	0.020	0.002	0.002	Remainder	

composites. They conducted analysis such as X-ray powder diffraction (XRD) and energy-dispersive X-ray spectroscopy (EDS) and confirmed the presence of Al, TiB_2 and carbon in Al6061– TiB_2 –graphite composite. There is an improvement in the tensile strength, ultimate tensile strength and elongation with the addition of TiB_2 and graphite.

Jayakumar and Rangarajan⁴ investigated silicon carbide particulate reinforced composite using vertical centrifugal casting process. They have observed porosity towards the inner zone of fabricated samples. Particulates were not dispersed properly and moved outward during the process. It was resulted in higher hardness of samples.

Muthazhagan *et al.*⁵ fabricated aluminium based metal matrix composite using stir casting process reinforced with graphite particulate. It was reported that addition of graphite reduces strength due to a poor interfacial bond between aluminium alloy and graphite.

Umanath *et al.*⁶ investigated the dry wear behavior of AA6061 Aluminium alloy, reinforcement with fine particulates of silicon carbide and aluminium oxide. They found that the wear decreases with increase in volume content of reinforcements (for the fixed size of SiC and Al_2O_3 particulates). The microhardness of the composite specimens measured after the wear test increases with the increase in volume content of the reinforcements. The width of the scratches decreases with increase in volume fraction of the reinforcements.

Selvam *et al.*⁷ tried to fabricate aluminium alloy AA6061 reinforced with fly ash particles by compocasting method and studied the effect of fly ash content on microstructure and mechanical properties of AA6061/fly ash AMCs. The addition of fly ash particles enhanced the microhardness and tensile strength of the AMCs. AA6061/12 wt% fly ash AMC exhibited 132.21% higher microhardness and 56.95% higher UTS compared to unreinforced AA6061 alloy.

Abolhasani *et al.*⁸ investigated the fabrication of aluminium alloy using forward the extrusion process. It was reported that the ductility and elongation were improved at elevated temperature due to an evolution of grain size. This is also considered as one of the best methods in the processing of aluminium alloys. Ezatpour *et al.*⁹ investigated aluminium based composite fabricated using stir casting process reinforced with

alumina oxides. It was reported that addition of reinforced particulates produces agglomeration and reduced strength at agglomerated zone. Also, it was reported that strength was improved by increasing stirring speed up to 300 rpm.

Shanmughasundaram and Subramanian¹⁰ investigated aluminium based graphite particulate composite using the stir casting and squeeze casting process. It was reported that wear behavior of graphite particulate composite was improved compared to aluminium alloy. Bhandare and Sonawane¹¹ reported about the stir casting process of aluminium based composites. It was noticed that strength of composite merely depends on the dispersion of particulates. The stir casting process was reported as simple and effective fabrication process to get a uniform dispersion of particulate reinforced composites.

Suresh and Sridhara¹² investigated the aluminium, silicon carbide and graphite reinforced composite. It was reported that an equal percentage of silicon carbide and graphite improved the friction characteristics and could be used in tribology applications.

Radhika *et al.*¹³ investigated the aluminium, alumina graphite composite using Taguchi method. It was reported that the formation of a protective layer of graphite improves the wear characteristics of composites.

Bhujang *et al.*¹⁴ investigated composites with nitride to improve the wear properties. It was reported that the heat treatment process for composites improved the wear characteristics. Suresh and Sridhara¹⁵⁻¹⁷ reported that silicon carbide particulates with aluminium, graphitic composites improve its wear behavior. The Addition of silicon carbide equivalent to graphite improves the behavior. Kumar and Murty¹⁸ investigated the grain refinement of 6061 alloy by the addition of titanium-carbide and titanium-borides. Mechanical behavior of alloy improves with the grain refinement by this addition.

Pathak *et al.*¹⁹ reported that addition of silicon carbide into the aluminium alloy reduced elongation. Also, the addition of reinforcement increased wear resistance. Akhlaghi and Pelaseyyed²⁰ reported the in-situ powder metallurgy process based combined with stir casting process to fabricate aluminium alloy composites. The reinforcement agglomeration was reduced using this process. It was reported that this process improves the material behavior in a cost effective way.

Pillai *et al.*²¹ reported about the fabrication of aluminium, silicon alloys with better material behavior. It was reported that reduction of turbulent mixing and introduction of vibration during solidification improves the density and strength of alloy combined with heat treatment.

Based on the literature, it was observed that stir casting processes is one of the widely used fabrication process of aluminium based composite. By maintaining the stirring speed and stirring duration, reinforcement can be dispersed uniformly in alloy matrix and produces better strength. The addition of uneven quantity of reinforcements like silicon carbide, graphite improves its hardness and reduces the ductility. Also, it was noticed that research on zirconium dioxide - based composite was limited and the potential of such composite yet to be explored.

The addition of ceramic particles like zirconium dioxide to this grade of aluminium alloy improves the stiffness and corrosion resistance, also reduces its weight by altering its density. Also, the reduced thermal expansion improves the dimensional stability of this material which makes it suitable for high precision applications. It can be a good replacement of commercially available 6061 grade of aluminium alloys. The potential of zirconium dioxide and particulate reinforced metal matrix composite needs to be explored to enhance the material behavior. In this research, zirconium dioxide particulate reinforced aluminium alloy metal matrix composite was fabricated using stir casting processes, and its behavior was analysed.

Experimental Procedure

MMC fabrication process

In this study, metal matrix composite samples were fabricated using stir casting process. 6061 grade aluminium alloy was used as the metal matrix and zirconium dioxide particulates were used as reinforcement. Samples were prepared with 4%, 8% and 12% weight ratio of reinforcement respectively. The typical stir casting setup is shown in Fig. 1. Aluminium alloy bar was chopped into smaller pieces and kept into a crucible. The crucible was heated above 1000 C using an induction furnace, such that the aluminium alloy was melted. Later, zirconium dioxide particulates with various weight percentages were added into molten aluminium alloy. The semi liquid composition was stirred about 10 min at 450 rpm constant speed to get a

homogenous distribution of particulates. Later, the semi liquid composition was poured into a cylindrical mould made for casting.

Result and Discussion

SEM micrographs

The microstructure and dispersion of particulate reinforcements in the MMC were analysed using scanning electron micrograph. The MMC samples were fabricated to the dimensions of 15 mm x 15 mm x 10 mm. The samples were polished using various grades of emery sheets ranging from 220-1200 grid size and mirror finish was obtained by polishing it with alumina solution. The polished samples were etched using Keller's agent as per standard procedures.

The scanning electron micrograph of all samples was given in Fig. 2. It shows uniform dispersion of ZrO_2 particulates in the 6061 alloy with 2%, 4%, 6%, 8% and 10% reinforcement respectively. It is noticed that the particulate agglomeration was reduced considerably. It makes sure that proper stirring was employed during fabrication of MMC. The random orientation of particulates provides uniform strength in as-cast material.

Hardness measurement

Rockwell hardness measurement was performed on fabricated MMC with various reinforcement weight percentages. MMC consists of a soft matrix alloy and hard ceramic reinforcement so selection of hardness measurement location is important. Measurement should be avoided on pure matrix alloy or reinforcement agglomeration zones. Hardness measurement was taken at various places of material and averaged values are plotted with 5% error bar in Fig. 3. It is noticed that hardness improves with an increase in reinforcement. The addition of ZrO₂ particulate attributes to increase in hardness of MMC.



Fig. 1 — Stir casting setup

From Fig. 3, it is observed that the HRC of the sample having 10% ZrO₂ inclusion is high, and the values have been found to increase due to the addition of SiC particles in the composites. Furthermore, to analyse the effect of ZrO₂ inclusion in different weight percentages, changes in the HRC values from one level to the next are compared and shown in Fig. 3. Significant improvement in the micro-hardness value has been observed in comparison. This indicates that the resistance behavior of the AA6061/ ZrO₂ composites against indentation has been drastically increased due to the addition of ZrO₂ particles.

Tensile strength

The mechanical strength like yield and ultimate tensile strength of fabricated MMC samples was analysed using tensile test as per ASTM-E8 standard. The significant contribution of reinforcement in the MMC can be analysed from Fig. 4. Figure 4 shows the experimental data of base metal, alloy with 0-10% reinforcement respectively. It is noticed that variation in load bearing capacity of MMC decreases with increase in reinforcement. The maximum breaking load of MMC improves by decreasing the weight percentage to 6% reinforcement. The maximum



Fig. 2-SEM analysis of AA6061/ZrO2 composite samples

displacement and displacement of breaking load of MMC reduces with increase in reinforcement. It loses its strength rapidly during plastic deformation due to ceramic particulate reinforcement.

From Fig. 4 it is observed that the tensile strength value of the samples initially increases after then slowly decrease the strength for inclusion of ZrO_2 . This result implies that the reinforcement of ZrO_2 in the AA6061 matrix results in an increase in its resistance capability against tensile force in the initial stage and decreases for additional ZrO_2 inclusion. Furthermore, to analyze the effect of ZrO_2 inclusion in terms of weight percent.

Changes in the tensile strength from one level to the next are compared and are shown in Fig. 4. A greater increase in the tensile strength value has been observed in the specimen having 6% ZrO₂ inclusion specimens. It has been observed that there is an initial increase in tensile strength, followed by a decrease in strength, as observed during comparison of the 8%, 10% inclusion specimens, and the strength at decreased. This phenomenon may be caused by the decrease in the ductility of the composite in microlevels localized near the ZrO₂ particles.

Tribology behaviour

MMC and wear surface

The wear resistance behavior of fabricated MMC was analysed using the pin-on-disc test as per ASTM G0099 standard. The pin samples were fabricated from the MMC cast billets with the dimensions of 8 mm diameter and 32 mm length. The rotating disc was fabricated using a hardened steel of 62HRC. The constant sliding distance of 1000 m was obtained by

maintaining the distance of the pin from the center of rotating disc and the sliding velocity.

A wear test characterizes the rate of material loss against the travel speed and time. It is measured by weighting the sample before and after the wear test. The wear characteristics merely depend on the surface hardness of the material that resists the loss of material when friction was generated by the pin on the sample's wear surface is given in Fig. 5.

The pin on disc wear test was carried out for various compositions of MMC and the wear rate for various compositions was calculated. Figure 6 shows the interpretation of variation in the wear rate for corresponding variation in the particulate ZrO_2 . The wear rate was directly proportional to the microhardness of MMC. The trend of plot clearly shows that the addition of ZrO_2 acts as a resistant to the MMC and reducing the wear. The ceramic reinforcement has major impact on the reduction in wear rate of MMC.



Fig. 3 — Microhardness value vs % of ZrO2 inclusion





Fig. 4 — Tensile strength value vs weight % of ZrO₂

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Sample 5. (8% ZrO2 and 92% AA6061)

Sample 6. (10% ZrO2 and 90% AA6061)

Fig. 5 — Wear SEM analysis of AA6061/ZrO₂ composite samples



Conclusions

Based on synthesis and material characterization, the following conclusions may be drawn:

Zirconium dioxide particulate reinforced aluminium alloy MMC was fabricated for various ratios. SEM micrographs show that the reinforcement dispersed into the alloy matrix uniformly. Proper stirring for a prolonged duration reduces agglomeration of particulates in matrix alloy.

Hardness measurement shows that addition of ceramic particulates improves the hardness with an increase in reinforcement content. This is due to the dislocation of matrix alloy was restricted by the ceramic particulates which improves the hardness.

It is noticed that the addition of zirconium dioxide, particulate reinforcement reduces the strength by increasing brittleness.

The dry sliding wear behavior of $AA6061/ZrO_2$ composite was evaluated using the pin-on-disc apparatus. To predict the wear rate of AA6061/0-10wt% ZrO₂ composite, worn surface of $AA6061/ZrO_2$ composite was examined by using SEM micrographs to identify the possible wear mechanism during dry sliding.

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