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Microstructure and mechanical properties of ZA27 based SiC reinforced composite processed by multi directional forging

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Keywords: ZA27, metal matrix composites, multi directional forging, ultrafine grains

Abstract

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Influence of multi directional forging (MDF) on microstructural and mechanical properties of ZA27/ SiC 5 weight percentage (Wt%) composites were investigated. Stir casting technique followed by squeezing process was adopted for synthesis of composite. MDF process was conducted at 100 °C and 200 °C upto strain of 0.54 and 1.09 respectively. Microstructure analysis was carried out using optical microscopy, scanning electron microscopy and energy dispersive spectrometry. SiC particles were fairly distributed and some clusters were also observed. Density of composite decreased with the reinforcement of SiC particles as compared with ZA27 alloy, however porosity which was existing as casting defect was reduced by MDF process. Average grain size of 200–250 nm and 1 μ m was achieved for MDF processed sample at 100 °C upto 3 passes and at 200 °C upto 6 passes respectively. Addition of SiC particles and adoption of MDF technique improved the vickers hardness of composites. Ultimate tensile strength of ZA/SiC composite has increased from 380 MPa to 395 and 432 MPa respectively with 54 and 37 percentage of elongation to failure. Improvement in hardness and tensile strength is due to strain hardening and grain refinement. Ductility of MDF processed ZA27/SiC composite is attributed to uniform distribution of ultrafine equiaxed grain and micro constituents.

1. Introduction

Severe plastic deformation (SPD) developed as the most successful technique in producing ultrafine grain materials. Severe plastic deformation achieved by introducing the large strain in metals. According to the Hall-Petch relationship ultrafine grained structures provide high tensile strength [1], these ultrafine grained metals and alloys has a more potential for engineering and industrial application [2]. MDF method was developed by Salishchev et al, during multiple passes without changing its cross sectional dimension the MDF process can introduce larger strain into the material. Change in deformation axis during MDF process from one pass to another pass promotes to develop uniform strain which results in homogeneous microstructure [3]. Hexagonal close-packed (hcp) metals and alloys are considered as significant class of engineering materials. Although single crystals of hcp metals deform extensively by slip deformation but polycrystals generally exhibit poor ductility. Zinc with higher content of Aluminum is widely used in making thrust washers, sleeve bearings, pressure tight housings and many structural applications. Members of the ZA cast alloys are ZA-8, ZA-12 and ZA-27 alloy [4]. These alloys combine hardness and high strength to give good bearing properties with good machinability and wear resistance often superior to standard bronze alloys [5, 6]. ZA-27 alloy has the lowest density and the highest strength out of ZA alloys series [7]. But in some application zinc-aluminium alloys properties restrict their use at elevated temperature (>100 °C). To overcome this drawback, one hopeful approach to improve the elevated temperature properties was reinforcing alloys with alumina particles and fibers, SiC fibers or particles, glass fibers etc [8,9]. Metal-matrix composites (MMCs) are the mixtures of pure metal or alloy with a known fraction by volume percentage or by weight percentage of reinforcement phase, usually ceramics. It has been demonstrated that addition of SiC, Al₂O₃, other carbides and nitrides into an alloy can produce substantial improvements in hardness, strength and wear resistance [10–14]. The amount of SiC particulate added into the



Table 1. Chemical composition of ZA27Alloy.

Elements	Aluminium	Copper	Cadmium	Iron	Manganese	Tin	Zinc
Percentage (%)	25–28	2.0-2.5	005–0.06	0.06-0.07	0.01-0.02	0.02-0.03	69.3–72.9

matrix metal can be varied from 2 to 30 vol%, depending on applications. Stir casting is widely used because of its simplicity and low cost. Stir casting followed by squeezing at certain pressure is useful in reducing the porosity level in cast materials which shows better properties as compared with gravity casting [15, 16]. Major attention is currently devoted to the study of the effects of mechanical working on MMCs. It has been revealed that some improvements in properties like strength and ductility can be observed in alloys and particle reinforced composites with plastic deformation processes, such as forging, extrusion and rolling [17, 18]. Several works have been reported to analyse the effect of SPD process on grain refine of ZA alloys. Sharath et al [19] studied the influence of MDF on the mechanical properties and microstructure of zinc aluminium alloy and achieved the fine grains around 2 μ m for 3 passes and around 1 μ m for 6 passes. Initial lamellar structure was gradually refined to a spherical shape and distributed more uniformly as number of passes increased, resulted in improvement of mechanical properties. Purcek, et al [20] investigate the effect of ECAP on the tribological properties and mechanical of Zn-40Al-2Cu-2Si cast alloy. It was observed that after ECAP process elongation percentage of the alloy was increased from 0.8% to 14.4% without a decrease in its strength. Processed samples showed better wear resistance on longer sliding distances, due to improved impact toughness and ductility of alloy as well as morphological changes. ZA27/SiC is considered to be brittle because of hcp metal matrix with hard SiC ceramic particle reinforcement. MDF process under plane strain compression condition could be used for further processing of cast ZA27/SiC composites. This deformation study on ZA27 alloy can give further insight to the deformation behavior of hcp materials in the presence of SiC particles. To the author's knowledge, little data are available on multi directional forging of ZA27/SiC metal matrix composite. Much improved strength with good ductility can be obtained after processing by MDF with reduction in weight of the ZA27 alloy by SiC particles reinforcement. Aim of present research is to evaluate the influence of MDF process on microstructure and mechanical properties (including particle distribution) of ZA27/SiC composites fabricated by stir casting technique followed by squeezing.

2. Materials used and experimental details

Materials used in fabrication of composites are commercially available ZA27 alloy and Silicon Carbide. SiC with an average particle size 15–30 μ m was reinforced in ZA27 alloy. Chemical composition analysis of ZA27 was carried out using optical emission spectrometer and the composition of ZA27 alloy is shown in table 1.

Stir casting technique was adopted for fabrication of this composite. In this technique dispersed phase (SiC particles) is mixed with ZA27 alloy by means of mechanical stirring. Figure 1 shows the schematic diagram of stir casting setup. A weighted quantity of matrix material was melted in graphite based crucible using resistance furnace. Temperature was gradually raised to 550 °C. Hexachloroethylene tablets were used in small quantity to degas the melt. This molten metal was stirred at a speed 600 rpm to create vortex. Stirrer blades were designed in such way that it creates vortex to attain good particle distribution in matrix. Reinforcement is preheated to a



Figure 2. Squeeze casting die.



temperature of 400 °C in order to enhance wettability with the metal matrix material [21]. Furthermore, particles preheating remove moisture and improve compatibility with molten matrix material during stir casting followed by squeezing. Reinforcing particles were added with a separate pouring attachment at the rate of 15–20 g min⁻¹ into the melt with constant stirring. Mixture was poured into the preheated metal die and squeezed at 80 MPa pressure and allowed to solidify at room temperature. Figure 2 shows a photograph image of squeeze casting die. Dimension of cast ingot was $60 \times 50 \times 60$ mm³ of block and machined to dimension of $30 \times 25 \times 30$ mm³ for further processing. Solutionizing heat treatment was carried out on ascast material at 365 °C for 5 h in muffle furnace and quenched in water [22]. Solutionizing temperature was selected from binary phase diagram of Zn-Al alloy for which a single phase supersaturated Al exists at 365 °C. As-quenched composites are subjected to MDF processing for different number of passes by using split type die as shown in figure 3. MDF processing with identical cross sections by the application of load using a punch, work piece is pressed in such a way that entire volume of work piece is confined within the die. Work piece is plastically deformed in nearly the same way of plane strain condition. Solutionized samples were subjected to MDF process at room temperature, 100 °C upto three and 200 °C upto six numbers of passes i.e., to a total cumulative strain of 0.54 and 1.09. The samples were heated to required temperature upto 20 min in the furnace. Equivalent strain of 0.18 is applied in every pass by pressing the sample down words. Equivalent strain is calculated according to the equation (1).

$$\varepsilon_{\rm e} = \ln \frac{{\rm h}_1}{{\rm h}_2} \tag{1}$$

Where h_1 and h_2 are initial and final height of the specimen. Later, specimen was processed for further passes by rotating the specimen to required axis and forged to the same strain as in previous passes. MDF processing method is symmetrically shown in figure 4. For one complete cycle to finish, sample has to be deformed in all three directions. Constant pressing speed of 0.5 mm m⁻¹ was applied in MDF technique for different passes to obtain consistent forged structure by using 40 ton hydraulic press. The lowest possible temperature is desired during compression to control the grain growth. Molybdenum disulfide (MoS₂) paste was used as lubricant to avoid frictional effects. XRD studies were carried out with Cu-K α radiation of 1.54 Å with diffraction angle (2 θ) range from 30° to 90° and at scanning speed of 2° per minute at 30 kV tube voltage and 20 mA current with Ni filter. Theoretical and experimental densities of the ZA27/SiC 5 wt% reinforced composites were compared with MDF processed samples. Experimental density was determined by density measurement kit (Contech-CAS-44) assisted with accurate weighing balance machine. Experimental density is calculated using equation (2) and theoretical density was evaluated by rule of mixtures concept. Percent porosity of composites was calculated from equation (3).





Figure 5. Tensile test specimen (All dimensions are in mm).

$$\rho^{\text{Expt}} = \frac{D}{D - I} \tag{2}$$

Where, $\rho^{\text{Expt}} = \text{Experimental density (g cm}^{-3})$, D = Dry weight of sample, I = Immersed weight of sample in water.

$$\text{%porosity} = \{(\rho^{\text{Theo}} - \rho^{\text{Expt}}) \div \rho^{\text{Theo}}\} \times 100$$
(3)

Where, $\rho^{\text{Theo}} = \text{Theoretical density} (g/cm^{-3}), \rho^{\text{Expt}} = \text{Experimental density} (g/cm^{-3}).$

For microstructure analysis samples were taken from centre area of the forged specimen parallel to the last forging axis. Samples were subjected to mechanical polishing and chemically etched using 2.5% Nital as etchant for unprocessed samples. Processed sample were etched by Palmerton reagent (CrO_3 -200 g, Na_2SO_4 -15 g, H_2O -1000 ml). Scanning electron microscope (SEM) images were obtained using JEOL JSM 6380LA equipment. SEM with EDX was used to confirm the presence of SiC particle in the cast composites. Average grain size was measured with the help of image J software using linear intercept method. Vickers micro hardness was measured at room temperature in the plane perpendicular to the axis of final forged samples under 1.961 N load and with a dwell time of 20 s. Tensile specimens were prepared in the direction perpendicular to the final forging axis. The tensile test was in accordance with ASTM E8 M standard with 4 mm gauge diameter and 16 mm gauge length with a cross head speed of 0.1 mm per minute using universal testing machine (AG–X plus TM 100 kN). Figure 5 shows a schematic diagram of tensile test specimens. Fractured surfaces of the tensile specimens were also examined using scanning electron microscope.

3. Results and discussion

ZA27/SiC 5 wt% composite were cast and solutionized at 365 °C for 5 h. This material is successfully processed by MDF at 100 °C upto three passes and 200 °C upto six passes respectively as shown in figure 6. However material failed when processed at room temperature, because Zn is the main alloying element with hexagonal close packed structure which possesses limited slip systems [23] and the presence of brittle and hard SiC particles further limits the plastic deformation of Zn based material at room temperature. Lower strain hardening and more number of slip systems are active for Zn based alloy at higher temperature. These are the factors responsible for successful processing of material at higher temperature.



Figure 6. ZA27/SiC composite processed by MDF (a) At room temperature (b) Upto 3 passes at 100 °C (c) Upto 6 passes at 200 °C.



3.1. XRD analysis

Diffraction peaks of present phases were individually indexed. XRD patterns of the ZA27 alloy after solutioning at 365 °C for 5 h and MDF processed at different temperature are shown in figure 7. In ZA27 alloy and its composite system, α (Al rich fcc), η (Zn rich hcp), ε (CuZn₄ hcp) phases are present and along with these, β (Zn rich fcc) phase is also present in solutionized condition. Intensity of α and η decreased after solutionizing. High temperature solution heat treatment lead to large extent of dissolution of η and α phases, This is due to natural againg at room temperature [24]. Presence of SiC particles in ZA27 matrix facilitates the transformation of SiC neighboured matrix by accelerating Zn and Cu diffusion [25]. Plastic deformation due to subsequent MDF process has initiated the decomposition of β phase. Therefore, the peak intensities for the β phase in XRD profile decreased while those of α and η phases intensity increased after MDF processing at higher temperatures. MDF processing leads to increase intensity of η and α phases by replacing β phase. Presences of ε phases can be observed in sample of MDF processed at 100 °C for 3 passes and by further processing at higher temperature the intensity of ε phase decressed. Intensity of α and η phase were same after processing by MDF process at 200 °C upto 6 passes. Higher temperature improves the ability of diffusion and this will initate the diffusion of elements from metastable state to stable state [4].

3.2. Density

Physical properties depend on mirostucture and in ZA-27 alloy the microstructure is a complex function of casting process and subsequent cooling rates [26]. Figure 8 shows the variations in theoretical and experimental density value with porosity percentage. Presence of voids or porosity in cast sample gives the considerable difference in experimental and theoretical density values. According to rule of mixture incorporation of lower density SiC particle (3.21 g cm⁻³) in higher density ZA27 alloy, results decrease in density of resultant composite. Discrepancy between theoretical and experimental density values were less in case of MDF processed samples, as the number of passes increased. Mechanical working such as MDF processing reduces porosity level in cast materials. Difference in the density of ascast and solutionized sample maybe due to the dimensional





change in ZA27 alloy during the solutionizing treatment and by conversion of metastable phases to stable phase, which are retained by non-equilibrium solidification and solid-state transformation.

3.3. Microstructure

Microstructure plays prominent role in the overall performance of the composites as well as alloys. Optical microstructure of ZA27 alloy and its composite in ascast condition shows presence of dendritic structure. Grain size of ZA-27 alloy is comparatively larger than that of the composite [27]. Composition of ZA27 alloy and distribution of SiC particles is confirmed by the EDX analysis, which is shown in figure 9. Some micro-constituents in the OM and SEM correspond mainly to the phases α -Al rich fcc phase, β -Zn rich fcc phases, ε -CuZn₄ hexagonal close packed phase, η -Zn rich hcp Phase, τ' -distorted bcc structure are shown in figure 10(a). Samples after the solutionizing treatment dendritic structure will break and forms the grain structure as shown in figure 10(b). Dendrites grow away from the particle, due to the restriction caused by the particle to solute enrichment [28]. At 365 °C, the dissolution of Zn and Al takes place. Supersaturated β phase in as-quenched specimen later transformed to α , ε and η phases. Figures 10(c), (d) indicates some cluster and fair uniform distribution of SiC particles in ZA27/SiC composite material for both ascast and solutionized condition.

It is clear from the optical microstructure of figures 10(e), (f) that there is a substantial grain refinement due to multidirectional forging process. Fine grains were seen in MDF processed samples for 3 passes at 100 °C and slightly larger grain size can be observed in MDF process sample for 6 passes at 200 °C. Surrounding area of SiC particle is completely filled by matrix material after the MDF process as compared with unprocessed composites and it is shown in figure 10(e). Porosity associated with particle clusters was reduced by MDF process. When ZA27 alloy is cooled from the melt condition to room temperature, it undergoes several phase transformations, like ($\alpha + L$), β , ($\alpha + \beta$), and finally ($\alpha + \eta$) [4]. The incorporation of low Cu content in the zinc aluminum alloy will lead to development of an inter-metallic compound CuZn₄ (ε) at 377 °C [15]. Microstructure of ZA27 as-cast alloy contains aluminium-rich matrix (α -fcc) and an interdendritic zinc-rich phase (η -hcp), (ε -CuZn₄),





(τ' -distorted bcc structure - Zn₁₀Al₃₅Cu₅₅). Light colored region is aluminium-rich (α) phase while the grey structure surrounding it is a mixture of zinc-rich (η) and aluminium rich phases. Dark regions are the zinc-rich (η) phase which is as shown in figure 11(c). Dendritic structure can be seen in initial as-cast material, after solution heat treatment and MDF processing, dendritic structure converts almost into agglomerates of Zn-rich and Al-rich phase with lamellar structure. Similar kind of observation are made by many researcher's on Zn base alloys[15]. A coarse-grained structure in unprocessed sample can be observed with the grain size of 20–25 μ m. Multi direction forging processed upto 3 passes at 100 °C shows ultrafine grain structure with an average grain size of 200–250 nm as shown in figure 11 (e₂) with corresponding grain size histogram, for convenience it is smoothed using gauss fit. After six passes of MDF at 200 °C, a homogeneous grain structure with an average grain size of approximately 1–2 μ m was achieved. At 200 °C upto 6 passes MDF processed sample shows larger grain size as compared with MDF processed samples at 100 °C upto 3 passes. It is due to grain growth in composite material processed at higher temperature [29]. Figure 11(f) shows ZA27/SiC MDF processed at 200 °C for 6 passes having the mixture of cellular and lamellar type of grain structure. Either eutectic or peritectic β phase transforms into lamellar α and η phases in style of cellular decomposition [25]. Formation of ultrafine grain structure were attributed to very large strain deformation.

3.4. Mechanical properties

Vickers hardness measured on the plane perpendicular to the axis of last compression axis of the MDF processed samples. Results of vickers hardness value are shown in figure 12. The hardness value of solutionized ZA27 alloy and ZA27/SiC composite increased from 117 to 134 and 148 Hv respectively. Hardness value increased to 170 and 166 Hv for 3 passes at 100 °C and 6 passes at 200 °C respectively. Presence of hard SiC particles in soft matrix





contribute to improve the hardness of composites. This improvement in hardness is also attributed to thermal strain mismatch between the SiC particule and ZA27 matrix during casting process. These thermal strains value decreases as the distance between reinforcement and matrix interface increases. These thermal strains are primarily located near the reinforcement-matrix interface [28]. Increase in hardness of processed material due to the grain refinement with increased volume of grain boundaries which occur during plastic deformation.

Figure 13 illustrates the engineering stress versus engineering strain for tensile test results of ZA27 ascast alloy and composite after solutionized, MDF processed up to three passes at 100 °C and up to six passes at 200 °C. The elongation to failure is a quantifiable measure of ductility, and is taken as the strain at which the sample breaks. Initially ascast and solutionized composite sample show lower percentage of elongation with an increment in ultimate tensile strength from 304 to 324 and 380 MPa respectively. However, after being processed by MDF technique, the strength and ductility were improved simultaneously. UTS and percentage of

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elongation for sample processed at 100 °C upto 3 passes is 395 MPa and 54%, respectively. Sample processed at 200 °C upto 6 passes showed ultimate tensile strength of 432 MPa and 37% of elongation. Repetitive pressing resulted in strain hardening which is the cause of dislocation multiplication. There is a clear enhancement in strength as dislocation density is increased and presence of SiC particles obstructs the movement of dislocation. Enhancement of ductility is due to uniform distribution of secondary phase particles, reduction in porosity, inhomogeneity and fine grain structure. Existence of SiC particles and the lamellar structure can hinder the dislocation movement, which inturn gives high strength in MDF processed samples. Presence of intermetallic copper rich ε phase, which is basically coarse and hard, which will effect in lowering the strength and ductility, this can be overcome by adoption of high temperature processing technique. However, it is possible that the lamellae may block the sliding of equiaxed grains, leading to higher strength in the processed sample. Spherical and well spread Al-rich and Zn-rich phase obtained provides more equiaxed grains as seen in SEM micrographs presented in figure 11(f). If the ultrafine grains are reasonably stable at elevated temperatures, there is a potential for achieving excellent superplastic properties [30].

Figure 14 shows the SEM micrographs of tensile fractured sample surface of ZA27 alloy and its composite in both forged and unforged conditions. Fracture of particle reinforced metal matrix composites is very much dependent on particle strength and reinforcement/matrix interface strength. If the strength of the interface bonding between reinforcement and matrix is greater than the praticle strength, then it leads to fracture of particles before the interface. Matrix void growth takes place and shear localization between fractured particles results in failure of the composites. Second scenario of damage is when interfacial bonding strength between the reinforcement and matrix from the particle strength which leads to void nucleation and growth at the interface, due to decohesion of the matrix from the particle [31, 32]. Ascast material of both reinforced and unreinforced samples followed brittle mode fracture because of its dendritic structure, this consequence in a higher tendency of crack nucleation and propagation. Precipitation of non-equilibrium hard ε phase also contributes to the brittleness of as-cast materials. Dendritic type structure contains interdendritic porosity





during solidification process due to incomplete filling of liquid melt is the cause for the brittle fracture in the ascast materials. Similar type of result was observed by other researches in SiC and B_4C reinforced composite materials [33–36]. In solution heat treated samples small amount of dimples were seen and it is shown in figure 14(h). For solutionized condition, fracture surface was composed of micro-porosities and dimples were larger in size so ductility was low. The presence of dimples shows that the mode of fracture is changing from brittle to ductile. And there is no much significant difference between figures 14(b), (d) it has almost same percentage of elongation. Micrographs of fracture durface at lower magnification is also taken at the equidistance to show the mode of fracture which is on the left side of every fractured microscopic image respectively. For MDF processed at 100 °C upto 3 passes is having finer grain size as well as dimple size as compared with samples of MDF processed at 200 °C upto 6 passes. This shows that material as completely converted from brittle mode to ductile mode of fracture and formed cup and cone type of failure which is clearly indicated in figures 14(i) & (k). After MDF processing dimples size decreased and showed a homogeneous distribution across the fracture surfaces. It resulted in improved ductility compared to solutionized material and this represents the material was deformed uniformly in the equiaxed structure with fine grain boundaries.

4. Conclusions

In present research work, ZA27 alloy as base matrix and SiC as reinforcement, the composites were fabricated using stir casting method followed by squeezing at certain pressure. Microstructural aspects and mechanical properties of base alloy, MDF processed and unprocessed composites were studied. Based on experimental evaluation, following conclusions can be drawn

- 1. Density of ZA27 alloy decreased by incorporation of SiC particles and porosity level decreased with increase in number of MDF passes. Some Clusters and fair dispersion of silicon carbide particles in ZA27 matrix were observed in microstructure and confirmed by EDX.
- 2. Multidirectional forging to a strain of 0.54 at 100 °C for 3 passes and 1.08 at 200 °C for 6 passes lead to the development of ultrafine grained structure in ZA27/SiC composite with an average grain size of 200–300 nm and 1–2 μ m, respectively. Grain growth phenomena was observed in MDF processed composites at higher temperature (200 °C) which leads to the formation of larger grains, even though composites were MDF processed for 6 passes when compared with 3 passes at 100 °C.
- 3. Elimination of the dendritic structure during solutionizing treatment promoted the increase in hardness for the ZA27 alloy and its composites, this is due to formation of equilibrium phases. The hardness value increased from 134 to 148 Hv by addition of hard SiC particle in soft matrix material, after MDF processing the hardness value increased to 170 and 166 Hv for MDF processed upto 3 passes at 100 °C and upto 6 passes at 200 °C respectively.
- 4. UTS was increased from 304 MPa to 324 MPa by solution heat treatment, incorporation of SiC improved UTS to 380 MPa with slight increase in ductility. After MDF processing at 100 °C upto 3 passes and 200 °C upto 6 passes, the UTS increased to 395 MPa and 432 MPa respectively. The highest ductility of 54% was obtained when the sample forged at 100 °C 3 passes, which is increased by more than 7 times in comparison with initial solutionized ZA/SiC composite samples. The excellent ductility attributed to its uniform distribution of secondary phase particles, reduction in inhomogeneity, porosity and fine grain structure.
- 5. Initial ascast condition showed brittle type of fracture. Brittle mode of fracture was transformed in to ductility mode by MDF processing at 100 °C upto 3 passes and at 200 °C upto 6 passes. Dimple type of fracture reveals the ductile behavior. After MDF processing dimples size decreased and showed a homogeneous distribution across the fracture surfaces.

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