Tensile and compression behaviour, microstructural characterization on Mg-3Zn-3Sn-0.7Mn alloy reinforced with SiCp prepared through powder metallurgy method

G Robert Singh, S Christopher Ezhil Singh, M Sivapragash, Lenin Anselm, R Sanjeev Kumar and A Haiter Lenin

Faculty of Mechanical Engineering, Swarnandhra College of Engineering and Technology, Narasapur, Andhra Pradesh, India
Faculty of Mechanical Engineering, VimalJyothi Engineering College, Chemperi, Kannur, Kerala, India
Faculty of Mechanical Engineering, universal College of Engineering and Technology, Tirunelveli, India
Faculty of Mechanical Engineering, Shinas College of Technology, Sultanate of Oman
Department of Mechanical Engineering, Kombolcha Institute of Technology, Wollo University, Ethiopia

E-mail: drahl Lenin@kiot.edu.et

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Abstract
In this research paper, Mg-3Zn-3Sn-0.7Mn/SiC composite is developed by reinforcing various weight fractions of SiCp in Mg-3Zn-3Sn-0.7Mn alloy through powder metallurgy route. The weight fraction of SiCp usage is varied from 3% to 15% in Mg-3Zn-3Sn-0.7Mn alloy (i.e., in Mg-3Zn-3Sn-0.7Mn/xSiC; the sample values are varies for x is 3, 6, 9, 12 and 15%). The effect of SiCp addition got tested against its tensile strength, compression behavior, hardness, microstructure, alloying nature and porosity. This study shows better grain refinement with improved properties while reinforcing Mg-3Zn-3Sn-0.7Mn alloy with 6 wt% SiC composites. It was observed that the grain refinement occurred while adding up to 6 wt% of SiC particles in the composite and thereafter increase in SiC caused little grain refinement effect. Hardness is getting increased with the increase of SiC weight fraction and reached maximum to 133 HV at 12SiC/Mg-3Zn-3Sn-0.7Mn. Higher UTs of 393 MPa obtained from the sample prepared with 12%SiC for 0.0533 s⁻¹ strain rate. The highest UCS of 341 MPa is obtained from the sample made with 15%SiC inclusion for 0.0533 s⁻¹ strain rate. From the SEM fracture analysis, the Mg-3Zn-3Sn-0.7Mn alloy and Mg-3Zn-3Sn-0.7Mn/SiC composite exhibit the almost same type of fracture called quasi-cleavage regardless of the % addition of SiC reinforcement. It was observed that the increase of SiC weight fraction increases the UCS because of its increased load-bearing capacity and reduction in cleavage facets.

1. Introduction
Magnesium (Mg) alloys and composites have various applications in an automobile field and in making of human implants because of its high strength to weight ratio and biocompatibility of magnesium [1, 2]. The success of these applications depends on the superior mechanical and physical properties shown from the studies carried out by many researchers; it is evident that the addition of Zn, Sn and Mn is come to improve the mechanical properties of magnesium alloy. Reinforcing of SiCp also drastically influence the mechanical properties. Some researches were extended with various magnesium alloys with composite nature developed through different processing [3–12]. The Mg-1.0Mn alloy by casting followed by hot extrusion obtained superior mechanical properties [13]. The high strength TAZ1031 Mg alloy at a ram speed of 0.1 mm/s and an extrusion temperature of 250 °C the same values were used for this study too [14]. The addition of Sn to the Mg-6Zn-1Mn alloy improved mechanical properties [15]. The effect of SiC particle reinforcement in AZ91 alloy and attained improved tensile and yield strength in AZ91/3SiC composite. SiCp reinforced in AZ31B alloy improves the mechanical properties and refined the grain size of AZ31B/SiCp composite [16, 17]. Taguchi based grey analysis
used to optimize the compositional elements of magnesium alloy to get better mechanical properties. The optimized alloying elements for Mg-Zn-Sn-Mn alloy used for this study are found from the previous research done by (Robert Singh et al 2014 and 2015). From the studies carried out by researchers, it is evident that the addition of Zn, Sn and Mn improved the mechanical properties of magnesium alloy [18]. Silicon carbide has high hardness, therefore the inclusion of SiC into the Mg alloy enhances it mechanical properties, and it was discussed by the researchers [19, 20].

In the present study, Mg-3Zn-3Sn-0.7Mn alloy is selected based on the previous studies (Robert Singh et al 2014 and 2015) and the vast applications (Kulekci et al 2008 and Gunde et al 2011). Towards further improvements in its mechanical properties it is proposed to reinforce through powder metallurgy route with Mg-3Zn-3Sn-0.7Mn/SiCp composite. The weight fraction of SiCp is chosen from 3% to 15% (i.e. in Mg-3Zn-3Sn-0.7Mn/xSiC (x = 3, 6, 9, 12 and 15)). The effect of SiCp reinforcing is analyzed against microstructural and mechanical properties.

2. Materials and Manufacturing

2.1. Materials
The Mg-3Zn-3Sn-0.7Mn/SiCp composite used for reinforcing is made with Mg-3Zn-3Sn-0.7Mn matrix and SiCp filler with an average particle size of 0.5 μm. The Mg-3Zn-3Sn-0.7Mn alloy is made with the metal powders having an average particle size of Zn 15 μm, Sn 40 μm, Mn 40 μm and Mg 60 μm. All these metal powders were mixed by powder blender and converted as Mg-3Zn-3Sn-0.7Mn powder alloy.

2.2. Fabrication of Mg-3Zn-3Sn-0.7Mn/SiCp composite
The samples were prepared with the five specified SiC weight fraction combinations; Mg-3Zn-3Sn-0.7Mn/3SiC, Mg-3Zn-3Sn-0.7Mn/6SiC, Mg-3Zn-3Sn-0.7Mn/9SiC, Mg-3Zn-3Sn-0.7Mn/12SiC, Mg-3Zn-3Sn-0.7Mn/15SiC respectively the powders in the above said weight percentage were mixed with Mg-3Zn-3Sn-0.7Mn alloy using RETSCH PM-400 alloying machine for 1 h at 150 rpm. After mixing, it got cold compacted using a hydraulic pressing machine into billets of 60 mm diameter with 35 mm height under 690 MPa pressure. Thereafter, the compacted composite alloy billets were heat treated at 400 °C in a muffle furnace over a period of 60 min

Then the prepared heat-treated billets were extruded to a 13 mm rod at 400 °C with an extrusion ratio of 25:1. Colloidal graphite was used as lubricant during hot extrusion to avert stick on effect. These extruded composite rods (Mg-3Zn-3Sn-0.7Mn/xSiC) were cut into rods of 150 mm length. Thereafter the internal induced stress got released by heat treating in a muffle furnace at a temperature of 260 °C over a period of 15 min. The entire residual stress removal takes place in argon atmosphere so that; the magnesium is protected against atmospheric corrosion.

2.3. Microstructure characterization
The optical microscopy is obtained from the samples by using an Optical Microscope (METSCOPE-I, MUC 500), JEOL-JSM-5610LV SEM analyzer is used for analyzing the tensile and compressive fracture surfaces. X-ray diffraction test (XRD) (PAAnalytical X’Pert Pro Powder X’Celerator Diffractometer) was carried out to study the microstructural elemental analysis and to identify the new phase formation of tested composite. Microstructure samples were prepared by polishing with the help of a disc polishing machine using sand papers with 500–2000 μm grit size and diamond paste of 0.2 μm. An etchant containing 6 gm picric acid, 10 ml H2O, 5 ml acetic acid and 100 ml ethanol was used to divulge the microstructure of Mg alloy and composites as suggested by (Wang et al 2013).

2.4. Mechanical properties characterization
Compression and tensile tests were carried out in an INSTRON servo-hydraulic controlled dynamic testing machine. Each alloy and composite samples tested with three strain rates which are 0.0533 s^-1, 0.016 s^-1 and 0.005 s^-1. Tensile testing specimens were prepared as per the ASTM E8M standard and compression testing specimens prepared as per the ASTM E9 standard. Using Archimedes principle, porosity and density of Mg-3Zn-3Sn-0.7Mn alloy and Mg-3Zn-3Sn-0.7Mn/SiC composites were measured. During the density and porosity measurement distilled water was used as immersion fluid. For measuring weight, an electronic balance of accuracy 0.0001 g was used. Micro-hardness (HV) was done for the alloy composites by using Digital micro Vickers hardness tester (HV 1000Z) with 500 g load, and 10 s dwell time. The measurements were done with an average of five repetitions taken from each point towards getting accurate hardness value.
3. Results and discussion

3.1. Optical microscopic characterization of Mg-3Zn-3Sn-0.7Mn/SiCp composite

The optical microscopic images are taken from the samples of Mg-3Zn-3Sn-0.7Mn alloy and Mg-3Zn-3Sn-0.7Mn/6SiC, Mg-3Zn-3Sn-0.7Mn/9SiC, Mg-3Zn-3Sn-0.7Mn/12SiC and Mg-3Zn-3Sn-0.7Mn/15SiC Mg composites. All the Optical Microscopic images captured were listed in figure 1. Figure 1(a) shows the microstructure of Mg-3Zn-3Sn-0.7Mn without SiC. This shows a uniform microstructure with no observed porosity. Figure 1(b) shows the microstructure of sample made with Mg-3Zn-3Sn-0.7Mn/3SiC added Mg alloy. Few SiC particles were observed in the surface and all particles were uniformly distributed over the matrix of advanced alloy. Agglomeration (Agglomeration is the common phenomenon of accumulation of SiC particles) SiC reinforcements were observed around the matrix grain boundary and some particles trapped inside the grains. The same kind of agglomerating regions around grain boundary were observed in all the increased SiC particle added samples (6SiC, 9SiC, 12SiC and 15SiC) and shown in figures 1(e)–(f) respectively. From figures 1(a)–(c), it was observed that, the grain refinement occurred while adding up to 6 wt% of SiC particles in the composite and thereafter increase in SiC caused little grain refinement effect and the same is shown in figures 1(d)–(f) respectively. This grain refinement reduction over a weight percentage addition of SiC is identified due to the impedes caused by the SiC particles in the grain growth during hot extrusion and possible occurrence of dynamic recrystallization (Shen et al 2015).

3.2. XRD Characterization of Mg-3Zn-3Sn-0.7Mn/SiCp Composite

Figure 2 shows the x-ray diffraction (XRD) patterns of Mg-3Zn-3Sn-0.7Mn alloy and Mg-3Zn-3Sn-0.7Mn/SiC composites. The XRD pattern of Mg-3Zn-3Sn-0.7Mn alloy without SiC addition reveals binary phases of Mg2Sn, MnSn2, MgZn2, Mg2Zn11, Mn3Sn, and Mn2Sn. Among these, Mg2Sn and MgZn2 phases were responsible for the improvement of tensile and yield strength of Mg-3Zn-3Sn-0.7Mn alloy through precipitation strengthening (Qi et al 2014). The XRD analysis done with the samples added with SiC mesh confirms the presence of Mg5Si6 and Mg2Si binary phases. This is an evident of interface reaction between magnesium matrix and the reinforced SiC particles at higher percentage of SiC mesh.

3.3. Density of Mg-3Zn-3Sn-0.7Mn/SiCp composite

Table 1 show the measured porosity and density results of Mg-3Zn-3Sn-0.7Mn alloy and Mg-3Zn-3Sn-0.7Mn/SiC composites. The percentage of Porosity presence in Mg-3Zn-3Sn-0.7Mn alloy got increased from 0.35% to
0.47% while adding 3SiC (wt%). This go on increasing while adding SiC further and reaches 0.65% while adding 15SiC. The reduced levels of measured porosity for the prepared Mg-3Zn-3Sn-0.7Mn alloy is confirmed from Optical image (shown in figure 1) itself. This reduced level of porosity caused mainly due to the extrusion process and high compacting pressure. While adding SiC, the density of the composite increased with the increased dense SiC particle weight fraction it will reduces the effect of compaction and it leads porosity formation.

### 3.4. Effect of hardness on Mg-3Zn-3Sn-0.7Mn/SiC\textsubscript{p} composite

Figure 3 shows the plot of micro-hardness values of Mg-3Zn-3Sn-0.7Mn alloy and Mg-3Zn-3Sn-0.7Mn/SiC composites (added with 3SiC, 6SiC, 9SiC, 12 SiC and 15SiC). Micro-hardness increased from 71 HV to 93 HV after addition of 3SiC with Mg-3Zn-3Sn-0.7Mn alloy. Hardness is getting increased with the increase of SiC weight fraction and reached maximum to 133 HV at 12SiC/ Mg-3Zn-3Sn-0.7Mn.

### 3.5. Effect ultimate tensile strength on Mg-3Zn-3Sn-0.7Mn/SiC\textsubscript{p} composite

Figure 4 illustrates the trend of Ultimate Tensile Strength (UTS) variation of composite without and with the addition of SiC alloy for 0.005 s\textsuperscript{-1}, 0.0167 s\textsuperscript{-1} and 0.0533 s\textsuperscript{-1} strain rates. UTS of Mg-3Zn-3Sn-0.7Mn alloy with 0%SiC addition is observed as 242 MPa to 259 MPa when the strain rate increased from 0.005 s\textsuperscript{-1} to 0.0167 s\textsuperscript{-1}. Further increase of strain rate to 0.005 s\textsuperscript{-1} increased the UTS to 277 MPa. While adding 3% of SiC, the UTS

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**Table 1. Density and porosity of Mg-3Zn-3Sn-0.7Mn alloy and Mg-3Zn-3Sn-0.7Mn/SiC composites.**

| Materials               | Theoretical density (g cm\textsuperscript{-3}) | Experimental density (g cm\textsuperscript{-3}) | Porosity (%) |
|-------------------------|-----------------------------------------------|-----------------------------------------------|--------------|
| Mg-3Zn-3Sn-0.7Mn        | 1.825                                         | 1.819 ± 0.002                                  | 0.35         |
| Mg-3Zn-3Sn-0.7Mn/3SiC   | 1.849                                         | 1.841 ± 0.002                                  | 0.47         |
| Mg-3Zn-3Sn-0.7Mn/6SiC   | 1.874                                         | 1.864 ± 0.002                                  | 0.54         |
| Mg-3Zn-3Sn-0.7Mn/9SiC   | 1.899                                         | 1.888 ± 0.002                                  | 0.57         |
| Mg-3Zn-3Sn-0.7Mn/12SiC  | 1.925                                         | 1.913 ± 0.002                                  | 0.63         |
| Mg-3Zn-3Sn-0.7Mn/15SiC  | 1.952                                         | 1.939 ± 0.002                                  | 0.65         |
value for all three strain rates increased. And then it is gone increasing with the addition of SiC upto 12% and then a slight decrease is observed with the addition of 15%SiC. Higher UTS of 293 MPa obtained from the sample prepared with 12% SiC for 0.0533 s$^{-1}$ strain rate. The formation of intermetallic phases like, Mg$_2$Si and Mg$_5$Si$_6$ and the grain refinement caused by the addition of SiC particles were the reasons for this improvement of mechanical properties. The intermetallic phase Mg$_2$Si has embrittlement in nature and thereby it improves the hardness and reduces the ductility of composite.

The effect of reinforcing of submicron size SiC particles (SiCp) in AZ31B on mechanical properties was studied by Shen et al. 2014 and the UTS of the 10 vol% SiCp/AZ31B composite increased to 340 MPa from 125 MPa of as cast matrix [5]. The effect of reinforcing of various volume fractions of 1 μm size SiCp in AZ31B alloy on mechanical properties were studied by Shen et al. 2015 and it was observed that the UTS values enhanced when the increase of SiCp in the composite with the decrease of elongation. Hot extrusion also one of the reasons for the increased strength by DRX and refined grain size for both AZ31B alloy and AZ31B/SiCp composites [19]. Nie et al developed a composite by reinforcing 1 μm size SiCp in AZ91 Mg alloy and the yield strength of the AZ91/SiCp composite increased with the increase of SiCp vol%. But the UTS values increased to 150 MPa from 125 MPa for the addition of 3 vol% SiCp and further decreased to 140 MPa when the SiCp content increased to 5 vol% [18]. By increasing the % SiC mesh in Magnesium alloy matrix improved the tensile strength of the composite upto an extent for different alloys [21–23]. In the present work, increasing the % SiC from 0 to 12% in
3.6. Micro structural characterization of Mg-3Zn-3Sn-0.7Mn/\textit{SiC\textsubscript{p}} composite after tensile testing

The SEM images of tensile fractured surfaces of Mg-3Zn-3Sn-0.7Mn alloy, and the Mg-3Zn-3Sn-0.7Mn/\textit{SiC\textsubscript{p}} composites made with the addition of 3\%\textit{SiC}, and 12\%\textit{SiC} were studied and the same is shown in figure 5. The fracture surfaces of these samples consist of cleavage facets (rivers like pattern), small dimples and cracks. It shows the alloy and composites initially underwent some plastic deformation and caused these dimples. Subsequent increase in load causes further crack expansion across the grains and thereby which causes the small-sized cleavage facets that leads to failure. The Mg-3Zn-3Sn-0.7Mn alloy and Mg-3Zn-3Sn-0.7Mn/\textit{SiC\textsubscript{p}} composite exhibit the almost same type of fracture called quasi-cleavage regardless of the % addition of \textit{SiC} reinforcement. The different strain rates were observed from the tensile fracture surface zones and are shown in figures 5(a)–(i).

3.7. Effect of ultimate compressive strength on Mg-3Zn-3Sn-0.7Mn/\textit{SiC\textsubscript{p}} composite

The Ultimate Compressive Strength (UCS) values obtained from the compression test were plotted and shown in figure 6. UCS of Mg-3Zn-3Sn-0.7Mn alloy without the addition of \textit{SiC} is increased from 300 MPa to 317 MPa when increasing strain rate from 0.005 s\textsuperscript{-1} to 0.0533 s\textsuperscript{-1}. Also, the results show an improvement in the strength while adding \textit{SiC} with the alloy. The highest UCS of 343 MPa is obtained from the sample made with 12\%\textit{SiC} inclusion for 0.0533 s\textsuperscript{-1} strain rate. Among all the six samples and all three strain rates studied, the UTS and UCS obtained were higher at 0.0533 s\textsuperscript{-1}.

The addition of \textit{SiC} particles in Mg alloy matrix improved the compressive properties of the Mg MMCs [21, 22]. Garcés \textit{et al} (2011) studied the influence of \textit{SiCp} reinforcement in AZ31 Mg alloy on compressive response synthesized by PM route followed by hot extrusion. The addition of \textit{SiCp} in AZ31 alloy led to the increase of UCS with the increase of vol\% of \textit{SiCp} and the stress strain curve shows similar trends for both alloy and composites [23]. In the present work, increasing the % \textit{SiC} form 0 to 12\% in magnesium alloy matrix there is an increase in the UCS value for all strain rates 0.005 s\textsuperscript{-1} from 300 to 325 MPa, 0.0167 s\textsuperscript{-1} from 305 to 332 MPa and 0.0533 s\textsuperscript{-1} from 317 to 343 MPa.
3.8. Micro structural characterization of Mg-3Zn-3Sn-0.7Mn/\text{SiC}_p \text{ composite after compression testing}

The SEM analysis was carried out for the samples without SiC and with SiC from 3\% to 12\%SiC addition. Samples made with 15\%SiC were not considered because of its decreased UCS. The SEM images of compression tested Mg-3Zn-3Sn-0.7Mn alloy without SiC, Mg-3Zn-3Sn-0.7Mn/3SiC composite and Mg-3Zn-3Sn-0.7Mn/12SiC composites with three strain rates were shown in figure 7.

After a compressive test the SEM image of Mg-3Zn-3Sn-0.7Mn alloy sample surface for 0.005 s\(^{-1}\) strain rate shown in figure 7(a) contains cleavage facets, micro cracks and de-cohered particles that lead to poor compression strength. While with the 0.005 s\(^{-1}\) strain rate the Mg-3Zn-3Sn-0.7Mn/3SiC and Mg-3Zn-3Sn-0.7Mn/12SiC...
composites shows disordered sharp cleavage facets shown in figures 7(b) and (c). With 0.016 s⁻¹ strain rate, the fracture surfaces of Mg-3Zn-3Sn-0.7Mn alloy contain sharp cleavage facets in the form of rugged in figure 7(d). Larger cracks and agglomerates with sharp cleavage facets observed in the fracture surfaces of Mg-3Zn-3Sn-0.7Mn/3SiC and Mg-3Zn-3Sn-0.7Mn/12SiC. Mg composites (figures 7(e) and (f)). The fracture surface with 0.0533 s⁻¹ strain rate of Mg-3Zn-3Sn-0.7Mn alloy contains cleavage facets, few micro cracks and agglomerated particle clusters (figure 7(g)). Cleavage facets became sharp when increasing SiC content in Mg-3Zn-3Sn-0.7Mn/ SiC composites with 3% and 12% were represented in (figures 7(h) and (i)). From the SEM fracture analysis, it was observed that the increase of SiC weight fraction increases the UCS because of its increased load-bearing capacity and reduction in cleavage facets. Also, further compression, the soft Mg alloy matrix and SiC interface leads to deformation due to concentrated loading and creates micro cracks in the surfaces.

4. Conclusions

• The adoption of hot extrusion followed by powder metallurgy synthesize of composite alloy reduces porosity and ensures high density.

• The UTS, UCS and hardness (HV) of the Mg-3Zn-3Sn-0.7Mn alloy is improved while converting into Composite by adding SiC mesh.

• The better mechanical properties of Mg-3Zn-3Sn-0.7Mn alloy made with Mg-3Zn-3Sn-0.7Mn/12%SiC mesh were observed with the strain rate of 0.0533 s⁻¹ than the other two strain rates of 0.0167 s⁻¹ and 0.005 s⁻¹.

• The SEM micrographs shows the fractured surfaces of the composites when tested against UTS shows a quasi-cleavage fracturesand for UCS shows a cleavage fractures.

ORCID iDs

A Haiter Lenin @ https://orcid.org/0000-0003-4212-9163

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