Mechanical and tribological performance of Al-Fe-SiC-Zr hybrid composites produced through powder metallurgy process

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Abstract

In this work a ternary Al-Fe-SiC metal matrix composites were reinforced using Zr particles through powder metallurgy process. The Al matrix and the reinforcements were mixed in high energy ball mill at a speed of 250 rpm over a period of 5 h so as to develop a homogenously dispersed composite material. The composite powders are then pressed at 500 MPa using hydraulic press. The compressed composite green compacts are then sintered at 500 °C for 2 h and allowed to cool under furnace atmosphere. The densities, micro hardness and compressive strength of Al-10Fe-10SiC-10Zr hybrid composites were investigated and reported. The composite materials were then pressured at 500 MPa using hydraulic press. The compressed composite green compacts are then sintered at 500 °C for 2 h and allowed to cool under furnace atmosphere. The densities, micro hardness and compressive strength of Al-Fe-SiC-Zr composites were investigated and reported. The composite materials were characterized using SEM, EDS and XRD. The density of Al-10Fe-10SiC-10Zr hybrid composites was found to be around 3.44 g cm⁻³. The Zr particles have influenced the micro hardness of the composite materials. The micro hardness of the Al-10Fe-10SiC-10Zr hybrid composites was found to be better compared to Al-10Fe and Al-10Fe-10SiC hybrid composites. The compressive strength of the Al-10Fe-10SiC-10Zr hybrid composites was around 205 MPa which is 44% higher than the Al-10Fe composite material. The porosity of the hybrid composites has reduced when compared to that of Al-10Fe and Al-10Fe-10SiC hybrid composites. The wear studies reveal that Al-10Fe-10SiC-10Zr bear out better wear resistance. The predominant wear mechanism was identified as adhesive wear followed by plastic deformation. This improved wear resistance was due to the formation of oxides layers such Al₂O₃, Fe₂O₃ and also due to the presence of AlFe₃ and AlₓZr₄ intermetallics.

1. Introduction

The utilization of hybrid composite materials as a replacement of conventional materials has increased drastically in many areas such as aerospace industries, automobile industries and also in various industrial applications where better mechanical, wear and corrosion characteristics are needed [1–7]. Therefore the main objectives of the development of hybrid composites are to develop materials with low density and better strength along with superior wear and corrosion resistance [8–10].

In the development of composite materials it is important to select the matrix materials, reinforcements, percentage of reinforcement and finally the method and production parameters as per the requirements. Now a day due to the economic considerations the industries are opting for low cost materials, in order to overcome high production cost. The most widely used matrix used reinforcements are Al₂O₃, TiO₂, SiC and graphite [11–19]. T Sathish Kumar et al investigated the wear behavior of AA6082 alloy reinforced with Y₂O₃ and graphite particles. The studies revealed that the hybrid composites have micro hardness which is 40% higher than that of base alloy [20]. T Sathish kumar et al also studied the effect of heat treatment on tribological properties of Al-7Si-ZrSiO₄ hybrid composites manufactured using stir casting process. The results revealed that the wear resistance of the hybrid composites is much superior to that of base alloy [21].

The other important factor to be considered is the wettability of Al matrix when fabricated using powder metallurgy process. The ceramic reinforcements such as Al₂O₃ and TiO₂ does not easily wetted as a result of
surface oxides on Al matrix. In order to improve the wettability of the Al based composites other reinforcements such as Fe, SiC, and Zr are added [22–27]. The addition of these reinforcements increases the mechanical properties as well as tribological and corrosion resistance properties of the composite material.

Another major area of concern is the uniform dispersion of reinforcements with the matrix materials. Even though there are various methods for fabricating Al based composites such as stir casting method, the major disadvantage was the lack of homogenous dispersion of the reinforcements as the result of agglomeration and cluster formation. The powder metallurgy is one among those methods by which uniform dispersion of reinforcements can be achieved. Moreover the powder metallurgy has been proven to be one of the cost efficient and most reliable methods for fabrication of high melting point materials [5, 6, 8, 26, 27]. There are many literatures based on light weight reinforcements so as to improve the mechanical properties of Al based composite materials; however there are very few studies based on high density hybrid reinforcements so as to improve the mechanical wear and corrosion characteristics of Al based composite materials.

Novelty of this work is to study the effect of Zr reinforcement on the Al-10Fe-10SiC hybrid composites. It obvious, that the addition of ceramic particles Such as SiC will improve the mechanical properties and wear resistant properties of the composites. But there will be some negative effects in terms of increase in porosity and thereby making the composites more brittle in nature compared to the base material. The addition of Zr as reinforcement might improve the ductile nature of the composites by reducing the porosity since Zr particles have a density of 6.49 g cm$^{-3}$. Further the Zr particles exhibit good mechanical hardness and better wear resistant properties even at high temperatures.

In this work various proportions of Al-Fe-SiC-Zr hybrid nanocomposites were produced using powder metallurgy process. The hybrid composites are then fabricated into 8 mm cylindrical pellets using high speed steel die. The compacted green pellets are then sintered using muffle furnace. The sintered composite pellets are subjected to mechanical characterizations such as density, microhardness and compressive strength. The wear resistance properties were studied using pin on disc apparatus. Thus the main objective of this work is to develop hybrid nanocomposite materials with superior mechanical and tribological properties that can be utilized in automobile, aerospace and other industrial applications.

2. Materials and method

2.1. Materials
The pure aluminum was used as the base material and the Fe, SiC and Zr are used as reinforcements in weight percentage. All the materials used in this research work are of research grade and of purity level 99.5% respectively. The figure 1 shows the Scanning Electron Microscope images of Pure Al, Fe, SiC and Zr. The micrographs were taken in Secondary electron mode operated at 10 kV. The morphology of pure Al resembles a flake like structure with an average particle size of 50 $\mu$m. The Fe powders were elliptical in nature with a particle size of 20 $\mu$m. The SiC and Zr powders were crystalline in nature and their particle size was found to be around 5 $\mu$m and 3 $\mu$m respectively.

2.2. Production of hybrid composite materials
The table 1 shows the various proportions of Al-10Fe-10SiC-Zr hybrid nanocomposites. The selected proportions of matrix and reinforcements are then fed into a high energy ball mill consisting of tungsten carbide balls. The ball milling process was carried out for 5 h at a speed of 250 rpm under the presence of toluene as a process control agent so as to obtain homogenous and reaction free hybrid composite materials. The homogenously mixed composite powders are then compacted using uniaxial hydraulic press at 500 Mpa so as to develop an 8 mm cylindrical green pellet. The green pellets are then sintered at a temperature of 500 °C for 2 h and cooled under furnace atmosphere.

2.3. Microhardness and density
The microhardness of the Al-10Fe-10SiC-Zr hybrid composites was carried out using Vickers hardness equipment at a uniform load of 1 kg. The dwell time for the entire process was maintained at 20 s. The results of the experiments represent an average of 10 measurements and the standard deviation values were reported. The density of the composite specimens after sintering process was measured using Archimedes principle and the relative density and porosity of the composite materials were calculated by the relations.

\[
\text{Relative Density} = \frac{1}{1 - \text{Porosity}}
\]

\[
\text{Porosity} = \frac{\text{Theoretical Density} - \text{Actual Density}}{\text{Theoretical Density}} \times 100
\]
2.4. Compressive strength
The universal testing machine UTM was utilized to study the compressive strength of Al-10Fe-10SiC-Zr composite materials. The 8 mm diameter composite pellets are compressed at a uniform and gradual speed rate of 5 mm min\(^{-1}\).

2.5. Microstructural characterization
The Scanning Electron Microscope (SEM) was used to explore the microstructures of the Al-10Fe-10SiC-Zr composite materials. The topographical characterization was carried out using Atomic Force Microscope (AFM). The XRD analysis was used to explore the chemical compositions present in the hybrid composites. The EDS analysis was used to confirm the presence of various elements in the hybrid composites.

2.6. Wear analysis
The Al-10Fe-10SiC-Zr hybrid composite pellets of 8 mm diameter and 30 mm long were used as test specimen. The DUCOM make pin on disc apparatus was used to perform wear test as per the ASTM-G99 standard. The wear analysis was carried out for various conditions say applied load, sliding distance and sliding speed. The tests were performed for five different trials for each specimen and the average values are tabulated. The composite wear specimens were weighed before and after the experiments using electronic weighing scale [28, 29].

3. Results and discussion

3.1. Characterization
The figure 2 shows the high resolution Fe-SEM of Al-10Fe-10SiC-Zr hybrid composites of varying Zr content at the magnification of 10,000 \(\times\) at an operating voltage of 10 kV. From the figure it can be understood that the reinforcements are uniformly dispersed into the Al matrix as the result of 5 h milling time. It can be observed that the average particle size of Al powder was also reduced considerably due to ball milling process. The figure 3 represents the EDS mapping of Al-10Fe-10SiC-10Zr hybrid composite powders. From the spectra it can be confirmed that the composite materials has the presence of Al, Fe, SiC and Zr content. Moreover there is also formation of AlFe\(_5\), Al\(_3\)Zr\(_4\) intermetallics and AlFe\(_2\)C compound and ZrO\(_2\) which can be inferred from the EDS mapping. The AFM image of Al-10Fe-10SiC-10Zr hybrid composite is shown in figure 4. From the image it can be understood that the there is uniform dispersion of reinforcements with the Al matrix and also it can be noted that there is formation of surface oxides due to the ball milling process. The x-ray diffraction spectra of Al-10Fe-10SiC-5Zr and Al-10Fe-10SiC-10Zr hybrid composites are shown in figure 5. The XRD analysis were carried out.
Table 1. Density and Microhardness of Al-10Fe-10SiC-Zr hybrid composites.

| S.no | Composition       | Composition notation | Actual density (g/cm\(^3\)) | Theoretical density (g/cm\(^3\)) | Relative density (%) | Porosity (%) | Micro hardness (HV) |
|------|-------------------|----------------------|------------------------------|-----------------------------------|----------------------|--------------|---------------------|
| 1    | Al-10Fe           | C1                   | 2.98                         | 3.22                              | 92.55                | 7.45         | 101                 |
| 2    | Al-10Fe-10SiC     | C2                   | 3.05                         | 3.27                              | 93.27                | 6.73         | 118                 |
| 3    | Al-10Fe-10SiC-2.5Zr | C3             | 3.14                         | 3.36                              | 93.45                | 6.55         | 120                 |
| 4    | Al-10Fe-10SiC-5Zr | C4                   | 3.23                         | 3.46                              | 93.36                | 6.64         | 132                 |
| 5    | Al-10Fe-10SiC-10Zr| C5                   | 3.44                         | 3.65                              | 94.25                | 5.75         | 135                 |
using Xpert-3 diffractometer (45 kV, 30 mA) with Cu anode ($\lambda = 0.15406$ nm). The XRD spectra exhibit the characteristic peaks of Al, Fe, SiC and Zr which confirms the uniform dispersion of reinforcements in Al matrix. The peaks at $2\theta = 39.5^\circ$, $44.25^\circ$, $65^\circ$, $77.25^\circ$ and $82.3^\circ$ confirm the presence of Al in composite materials as per the JCPDS No: 34-0529, 06-0696. The characteristic low intensity $2\theta$ peaks at $37.5^\circ$ and $82.3^\circ$ corresponds to SiC which authenticates its presence in the composite materials (JCPDS No: 42-1172). The peaks at $2\theta = 44.25^\circ$, $65^\circ$ and $82.3^\circ$ also prove the presence of Fe particles in the composite materials (JCPDS No: 45-1203). The $2\theta$ peaks at $77.25^\circ$, $14.5^\circ$, $35.87^\circ$, $60.14^\circ$ are the characteristics peaks of Zr (JCPDS No: 41-0814). The formation of AlFe$_3$, 

![Figure 2](image_url)
Figure 3. Field Emission Scanning Electron Microscope (FE-SEM) mapping of Al-10Fe-10SiC-10Zr hybrid composites.
Al3Zr4 intermetallics and AlFe3C compound were observed from the XRD analysis (JCPDS No: 45-1203, 41-0814).

3.2. Density and micro hardness
The density, Relative density, porosity and microhardness of the Al-10Fe-10SiC- Zr hybrid composites are represented in table 1. The relationship between density and porosity of Al-10Fe, Al-10Fe-10SiC and Al-10Fe-10SiC- Zr hybrid composites are shown in figure 6. The density of the Al-Fe-SiC ternary composites has improved with the addition of Zr reinforcements. The density of the Al-10Fe composites was found to be 2.98 g cm⁻³ whereas; the density of the Al-10Fe-10SiC-10Zr hybrid composites has increased to 3.44 g cm⁻³. The porosity of the composite materials decreased with increase in Zr addition. The Al-10Fe-10SiC-10Zr hybrid composites have better density and porosity compared to other combinations. The reason behind this decrease in porosity is due the high density Zr reinforcements and also due the compaction pressure. It was also found that theoretical density of the composites was higher than the actual density of all compositions. The relative density percentage of the Al-10Fe-10SiC-10Zr composites was 94.25% and has increased 1.8% when compared with Al-10Fe composites. The microhardness of the Al-10Fe-10SiC-Zr hybrid composites has increased slightly with the increases in Zr addition. The improvement in microhardness was also due to the reduction in porosity of the composite pellets and also due to the formation of AlFe3, Al3Zr4 intermetallics.
3.3. Compressive strength
The compressive strength of various Al-10Fe-10SiC-Zr hybrid composites is shown in figure 7. The Compressive strength of the Al-10Fe-10SiC-Zr hybrid composites shows betterment with the increase in load bearing Zr reinforcement [30]. The other major reason for the improvement in compressive strength was due to formation of hard AlFe3 intermetallics and oxides as the result of sintering operation. The presence of SiC particles in the composite materials also played a major role in the improvement of compressive strength.

3.4. Wear analysis
The primary concern for any light weight material is long service life and less replacement period, thereby reducing the total expenditure incurred. Hence it is desirable to develop a material which has very less wear loss under sliding wear conditions. The effect of Zr reinforcement on the wear loss of Al-10Fe-10SiC ternary composites is shown in figure 8. From the figure 8(a) it can be understood that the increase in applied load has resulted in increased wear loss irrespective of Zr reinforcement. This phenomenon was due to the increased contact surface between the specimen and rotating disc. Figure 8(b) reveals that the wear loss of the composite materials increases as the distance of sliding increases. The increase in sliding distance increases the contact period of the composite materials with the mating surface thereby increasing the temperature at the interface. The increase in surface temperature further results in softening of materials and the deformation of materials takes place. From the figure 8(c) it is clear that the increase in temperature at the interface due to the increase in Sliding speed has resulted in softening of the composite pellet thereby increasing the rate of wear loss. It can be
also noted that the wear loss of Al-10Fe and Al-10Fe-10SiC hybrid composites are higher than the Al-10Fe-
10SiC-10Zr hybrid composites under all sliding wear conditions. The Coefficient of friction analysis of various
Al-10Fe-10SiC-Zr hybrid composites is shown in figure 9. The addition of Zr reinforcements has resulted in
reducing the COF values Al-10Fe-10SiC ternary composites. The coefficient of friction of Al-10Fe-10SiC-10Zr
composites has improved compared to that of Al-10Fe-10SiC-5Zr, Al-10Fe-10SiC-2.5Zr hybrid composites as

Figure 8. Wear loss of Al-10Fe-10SiC-Zr hybrid composites.
well as Al-10Fe and Al-10Fe-10SiC composite materials. This improvement in wear and friction characteristics of the 10Fe-10SiC ternary composites was due to the formation of AlFe$_3$ and Al$_3$Zr$_4$ intermetallics which improved the density and surface hardness of the composite materials. The other major reason was the formation of AlFe$_3$C compound which increases the hardness and self-lubricating property of the composite materials. This improvement in wear and friction characteristics of the 10Fe-10SiC ternary composites was due to the formation of AlFe$_3$ and Al$_3$Zr$_4$ intermetallics which improved the density and surface hardness of the composite materials. The other major reason was the formation of AlFe$_3$C compound which increases the hardness and self-lubricating property of the composite materials.
Figure 10. [A] FESEM image of Al-10Fe-10SiC-10Zr worn out Sample, [B] EDAX Spectra of Al-10Fe-10SiC-10Zr worn out Sample [C] SEM image of Al-10Fe worn out sample [D] SEM image of Al-10Fe-10SiC worn out Sample.
Further there is also formation of ZrO₂, Al₂O₃ and Fe₂O₃ tribo layers which also played a vital role in improving the sliding wear properties of Al-10Fe-10SiC-Zr nanocomposites. The figure 10 exhibits the high resolution FESEM image and EDAX spectra of Al-10Fe-10SiC-10Zr hybrid composites worn out surface after wear analysis. From the FESEM it is evident that the main wear mechanism was adhesive wear with micro cracking which leads to plastic deformation. Figure 10(B) represents the EDAX spectra of Al-10Fe-10SiC-10Zr hybrid composites after wear test, which confirms the presence of Al, Fe, SiC and Zr along with the presence of oxides such as ZrO₂, Al₂O₃ and Fe₂O₃ at contact surface. Figures 10(C) & (D) shows the SEM images of worn out surfaces of Al-10Fe composites and Al-10Fe-10SiC hybrid composites after wear test. From the spectra’s it can be confirmed that the Al-10Fe-10SiC hybrid composites has experienced abrasive wear along with delamination. Whereas the Al-10Fe-10SiC hybrid composites experiences abrasive wear followed by adhesive wear which leads to plastic deformation [31, 32].

4. Conclusions

The Al-10Fe-10SiC-Zr hybrid composites were produced through mechanical alloying process. The mechanical, tribological and corrosion resistance properties of the composites were studied at different conditions.

- The density of the Al-10Fe-10SiC-10Zr hybrid composites has improved to 3.44 g cm⁻³ from 3.14 g cm⁻³ for Al-10Fe-10SiC-2.5Zr hybrid composites.
- The Microhardness of the Al-10Fe-10SiC-10Zr (135 HV) hybrid composites is better to that of Al-10Fe-10SiC-2.5Zr (120 HV) hybrid composites due to the formation of AlFe₃C compound.
- The porosity of the Al-10Fe-10SiC-10Zr (5.75%) hybrid composites has reduced compared to that of Al-10Fe-10SiC-2.5Zr (6.55%) hybrid composites.
- The compressive strength of the the Al-10Fe-10SiC-10Zr hybrid composites has found to be better compared to other combinations.
- The wear resistance and coefficient of friction of the Al-10Fe-10SiC-10Zr hybrid composites has improved significantly compared to other combinations due to the formation of Al₃Zr₄, AlFe₃ intermetallics.
- From the findings of this study, it can be concluded that the Al-10Fe-10SiC-10Zr hybrid composites has better mechanical and tribological properties.

Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

Declaration of Conflicting Interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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