Experimental Investigations on Microstructure, Mechanical and Wear Behaviours of Al 7075-Boron Carbide-Cow Dung Ash Hybrid Metal Matrix Composites

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Keywords : hybrid aluminium metal matrix, Microstructure, Mechanical and wear properties.

ABSTRACT

This work focuses on the effects of reinforcements in microstructure, mechanical and wear properties when compared to aluminium 7075. Stir casting technique was used were boron carbide (B₄C) and cow dung ash (CDA) are reinforced by varying their percentage. Micro structural analyses were investigated by optical microscope. Mechanical and wear testing was carried out and fracture mechanisms for tensile and wear were analysed by scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX). Micro structural images reveal uniform distribution of reinforcements in the matrix. The mechanical and wear properties have increased in addition of B₄C - CDA particles in the matrix. Dimples, transgranular cleavage facets and micro ploughing, micro cuttings are revealed from the fractured specimens of tensile and wear respectively.

INTRODUCTION

Many manufacturing industries are extensively using aluminium and its alloys. Aluminium is chosen mainly because of their light weight construction. Usually, the mechanical and tribological properties of aluminium alloys can be improved in many ways, comprising of, (i) adding insoluble reinforcement to form Metal Matrix composites (ii) by precipitation hardening, (iii) by cryogenic treatments, (iv) by surface coatings etc.

Paper Received September, 2019. Revised December, 2020. Accepted December 2020, Author for Correspondence: Manikandan Rajan Among the above processes, metal matrix composites have achieved much attention in enhancing the properties of aluminium (Surappa, M. K. et al. 2003; Akhil, K. T et al. 2013; Mohan, K., et al. 2016; Baradeswaran A et al. 2013; Manikandan R et al. 2019).

Generally, a composite material consists of two or more insoluble phases, and their properties might be greater than that of its constituents. Because of its good engineering properties, easy fabricability and low density, aluminium is preferred to be as matrix material in most circumstances. The fabrication of aluminium composites are mostly done by (i) solid state processing (ii) Liquid state processing (stir casting, pressurized die casting, infiltration process) (iii) In situ processing. Among these methods, researchers have found that stir casting is the most profitable and hopeful route. Stir casting is preferred because of its vital parameters like stirring speed, stirring time, stirrer location, movement of the stirrer, holding time, melt temperature, design, preheating of die and reinforcements etc. can be considered. The optimum selection of these process parameters can decide the properties of composite material (Ramnath, B et al. 2014; Hashim, J et al. 2012).

In Aluminium Metal matrix composites, properties of matrix material (i.e aluminium) are enhanced by adding single hard reinforcement. The reinforcement can be continuous type (carbon fiber, SiC fiber etc) or discontinuous type (short fibers, whiskers and particles). Particle reinforcements in aluminium matrix composites are widely used. The ceramic particles like carbide (SiC, B₄C, TiC etc), borides (TiB₂,AlB₂ etc and nitrides – BN, AlN etc) and oxides (Al₂O₃, MgO, ZrSiO₄, ZrO₂ etc), are reinforced with aluminium melt (Radhika, N et al. 2017; Rao, V et al. 2014; Manikandan, R et al.2019). Addition of these reinforcement improves the mechanical and wear properties of aluminium and thus makes it suitable for numerous fields of application. Hybrid aluminium metal matrix composites enhance the properties of composites, than single reinforcement. Hybrid metal matrix is adding two or more synthetic

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ceramic in aluminium matrix (Bobić B et al. 2010; Alaneme KK et al. 2011; Yigezu et al. 2013). The current scenario in hybrid composites is reinforcing an agro waste with synthetic ceramic. Agro waste like bamboo leaf ash, rice husk ash, palm kernel shell ash, bagasse ash, corn cob ash, maize stalk ash, bean shell waste ash etc are researched. The benefits of adding agro waste are ease of access, low density, low cost and reduced pollution (Fatile et al. 2014; Bodunrin et al. 2016; Loh, Y. R et 2013; Aigbodion, V. S., et al. 2010; Arulraj, M. et al. 2018).

The elements present in cow dung ash are SiO₂, Al₂O₃, Fe₂O₃, MgO etc. which could enhance the properties of aluminium. Hence an attempt has been made in adding the same has second reinforcement. Aluminium with B₄C exhibit high hardness, good impact and wear resistance, low thermal conductivity, low specific density, and high stiffness. Aluminium 7075 is widely used in aeronautical applications for its high tensile strength. In this study the percentages of reinforcements are varied and the effects are evaluated through hardness and tensile strength. The fracture mechanism for tensile specimens is analyzed. Dry sliding wear tests are conducted on the fabricated specimens and surface morphologies for wear are analyzed. The distributions of reinforcements are evaluated with optical microscope and fracture mechanisms are evaluated by SEM with EDX images.

EXPERIMENTAL PROCEDURE

Selection of materials

Aluminium 7075 alloy is selected as the matrix material for the current study. The chemical composition of Al 7075 after spectro analysis is given in Table 1. The reinforcements selected for the study are boron carbide and CDA. CDA is thoroughly dried in sunlight and then burned to convert it into ash. To identify the chemical composition of CDA particles XRF (X-ray fluorescence – (Bruker S8 Tiger)) is used. The elements present in CDA particles are given in Table 2. The particle size of boron carbide is ranging less than 10 μ m, and CDA ranging less than 15 μ m are used in the study. Figure 1 (a & b) shows the SEM images of cow dung ash and boron carbide respectively.

Table 1. Chemical elements of Al 7075

Element	Zn	Cu	Mn	Mg	Fe	Cr	Ti	Si	Al
Ingot (wt%)	5.4	1.42	0.12	2.42	0.42	0.21	0.11	0.13	Bal.

Table 2

XRF element analysis for CDA

Si	K	Ca	Р	Mg	Al	Na	Fe	S
78.74	6.05	4.95	3.74	3.49	0.44	0.39	0.31	0.20

Fabrication process

The Al- B₄C -CDA hybrid metal matrix composite was melted in an electrical furnace. The melting temperature of 800°C is selected and samples are casted by for the current study. The schematic view of stir casting is shown in Figure 2 and process parameters selected for stir casting is given in table 3. A muffle furnace is used to preheat the reinforcements



WD = 4.6 mmb)SEM of B₄C particles

EHT = 5.00 kV

Fig.1. (a & b) SEM images of the CDA and B4C

Mag = 15.00 K X

Signal A = InLens

for two hours to remove the surface absorbed volatile contents. Three different percentages of reinforcements are selected and are given in Table 4. Once the melt has reached 800°C, the reinforcements are added in the liquid melt and a mechanical stirrer with four blades rotating in a range of (300 - 400 rpm)is used to stir for producing homogenous composite material. To elevate the solidification rate a stainless steel mould is used.

Micro structural analysis

The specimens of dimensions 10mm x 10mm

x 10mm was machined and polished with different grades of emery sheets for obtaining a clear surface for microstructural analysis. The polished specimens were etched with Keller's reagent as per standard metallographic procedure.



Fig.2. Schematic view of stir casting setup

Hardness test

The hardness tests were conducted as per ASTM: E10 standard. The samples were machined and tested for hardness in Brinell scale; with 2500 N load and 5 mm diameter steel ball indenter for 30 seconds. Three indentations were taken at room temperature and the average value was calculated.

Table 3. List of process parameters selected for stir casting process

Process parameters	Selected parameters		
Processing Temperature	1000°C		
Preheat temperature of mould	300°C		
Reinforcement particle preheat temperature	400°C		
Stirring speed	300-400 rpm		
Stirring time(min)	3		
Blade Angle	45°		
No of Blades	4		
Position of Stirring in the melt	Up to ³ ⁄ ₄ depth		

Table 4. Percentage of reinforcements

Sample	Al 7075 (Weight %)	B ₄ C	CDA
A 0	100%	0%	0%
A 1	90%	2.5%	7.5%
A 2	90%	5%	5%
A 3	90%	7.5%	2.5%

Tensile Test

The tensile test was performed using M 30universal testing machine. Two specimens were prepared as per the ASTM: E08 standard which is shown in figure 3. The average value of ultimate tensile strength was calculated.

Dry Sliding Wear test

The experiments were conducted according to ASTM: G99 standard. The fabricated specimens were tested for dry sliding wear using Pin-on-Disk apparatus. Three constant process parameters are selected to conduct wear experiments namely, sliding velocity (2.997 m/s), load (30 N) and time (15 min.). Cylindrical specimens of diameter 10 mm and height 28mm is prepared for studying wear resistance.

RESULTS AND DISCUSSION

Evaluation of microstructure

The microstructure images for the reinforced sample A0 and A3 are shown in Figure 4 (a and b). The optical images reveal that uniform distribution of reinforcements is achieved. During solidification of 7075 aluminium alloy the formation of intermetallic phases such as MgZn₂, Al₂Mg₃Zn₃, Al₂CuMg, Al₂Cu, Al₁₃Fe₄ and Mg₂Si occurs.



Fig.4 (a). Microstructure for sample A0



Fig.4 (b). Microstructure for sample A3

From the figure for sample A0 the micro segregation of $MgZn_2$ with fine grains is uniformly distributed in aluminium alloy. Dislocation movement takes place across the grain boundaries from one grain to another. Thus the properties of the material depend

on increased grain boundaries which can be achieved by addition of reinforced particles. The reinforced particles act as a barrier to the dislocation movement of grains resulting in increasing the ultimate tensile strength. Selection of process parameters for stir casting acts as a vital part in homogeneous distribution of reinforcements in the matrix.

Evaluation of Hardness

Addition of B_4C and CDA particles in aluminium matrix increases the mechanical properties for the fabricated hybrid composites. The graphical representation on the effect of reinforcements on hardness is shown in Figure 5 and the standard deviations of hardness are given in Table 5. The hardness values have gradually increased with different percentage of reinforcements.



Fig.5. Effects of B₄C and CDA on Hardness

 B_4C is well known particle for its hardness and its presence is one of the reasons for increase in hardness for all samples. Sample A1, having 7.5% CDA particles showed higher hardness (118 BHN) than sample S0 (110 BHN). The hardness has slightly increased when compared with sample A0. While increasing the CDA particle in the matrix the hardness value reduces, this may be due to the presence of refractory elements (SiO₂, Al₂O₃ and Fe₂O₃). Increasing the CDA particles in the matrix leads to assist in movement of fine CDA particles easily causing slip during indentation which causes decrease in hardness value. The density difference between the reinforced particles and aluminium matrix may also be the reason for minor change in hardness for sample S1.

Even percentage of B_4C and CDA particles are added in sample A2. The presence of B4C has restricted the flow movement of fine particles of CDA and increase the hardness more when compared to sample A1. The percentage of B_4C is more for sample A3 and hence during indentation, resistance to plastic deformation occurs which hardens the surface area by increasing the hardness. Aluminium being a ductile material turns brittle when hard ceramics are added in the matrix which enhances the hardness. Uniform distribution of reinforcements, rate of solidification, density of reinforcement particle and less porosity are few major parameters influencing the hardness of composites (Alaneme K. K. et al. 2013). The resistance against indentation on the fabricated hybrid composite samples were identified by Brinell hardness test. The average values of hardness are found to be 110, 118, 132 and 144 BHN for A0, A1, A2 and A3 samples respectively. The highest value was found in sample A3 having 7.5% B_4C and 2.5% CDA of reinforcements and 30.90% of increment was achieved when compared to base alloy.

Sample	Brir	nell Hard (BHN)	ness	Average Hardness	Standard Deviation for Hardness	
~	Trial 1	Trial 2	Trial 3	value		
A 0	109	110	112	110.33	1.53	
A 1	115	119	120	118	2.65	
A 2	133	131	133	132.33	1.15	
A 3	142	144	145	143.67	1.53	

 Table 5. Mean and Standard deviation for hardness

Evaluation of Tensile properties

The graphical representation of the average tensile strength is shown in Figure 6 and the standard deviations of tensile strength are given in Table 6. It is observed that the tensile strength has increased for all the composite samples when compared with Al 7075. Inclusion of B₄C particles increases the tensile strength in the composite samples. Addition of B₄C particles causes brittleness in the composites which in turn increases the strength in composite material. Reinforcing the particles has increased the tensile strength of composite material by 56%. The hard nature of B₄C and CDA acts as a barrier and restricts the crack propagation to generate which leads to dislocation of matrix, also called as Orowan mechanism. The increase in strength is achieved only when the dislocation density in the matrix increases and hence it is proved that the reinforcements have adequate impact in the fabricated hybrid composite material. The grain boundaries play a vital role in composite materials. The strength and restricted growth of micro cracks are achieved only with increased grain boundaries. Addition of CDA and B4C has increased the grain boundaries in the matrix.



Fig.6. Effects of B₄C and CDA on Tensile strength

Dispersion of reinforcements uniformly and interfacial strength in the aluminium matrix can be attained by preheating the CDA and B4C particles during casting process. The preheating provokes the reinforcements in generating the thermal stress which may influence the strength in composite materials. The thermal mismatch between B₄C-CDA and the matrix is also one of the reasons for increase in tensile strength. It is well known that the thermal expansion coefficient of B4C particle is 5x10-6/°C whereas for aluminium alloy is 23x10-6/°C. The uniform dispersion of B_4C and CDA particles without any flaws and defects produces excellent bulk mechanical properties. It is also clear from the result that the fabricated hybrid composite materials induce high strength to Al 7075 alloy and increased the resistance to tensile stresses leading to superior ultimate tensile strength (Alaneme K. K. et al. 2013). The resistance against tensile force on the reinforcements can be identified by the ultimate tensile strength value. The average tensile strength values are found to be 184.8, 249, 265 and 288 MPa for A0, A1, A2 and A3 samples respectively. The highest value was found in sample A3 having 7.5% $B_4C - 2.5\%$ CDA composition and 56% of increment was achieved when compared to base alloy.

Table 6. Mean and Standard deviation for tensile strength

	Tensile	strength	Average	Standard Deviation	
Sample	Trial 1 Trial 2		Tensile strength	for Tensile strength	
A 0	186	183.7	184.8	184.85	
A 1	250	248	249	249	
A 2	263	267.3	265	265.15	
A 3	289.4	287.2	288	288.3	

In tensile test specimens the fractures are categorized into two, namely brittle and ductile fracture. In fracture mechanism, two major factors which influences are the non-uniform distribution of reinforcements in aluminium alloy metal–matrix and the formation of second phases during the casting process respectively. The first factor attributes the difference in strain carrying capability between the hard and brittle reinforcement (B₄C-CDA) and the soft and ductile aluminium alloy metal – matrix. The second factors are 7075 aluminium alloy matrix, differences in the coefficient of thermal expansion among the reinforcement particles, and the precipitated phases.

The tensile fracture analysis is done to identify the failure patterns namely transgranular cleavage facets and microvoid coalescence through sem images. In hybrid composite materials the brittle fracture occurs due to very less plastic deformation which are patterned as transgranular cleavage facets. The ductile fracture occurs during necking in specimens which are patterned as microvoid. The transgranular facets tend to increase crack propagation through grains and microvoids combine together and tend to crack when load is greater than the tensile strength. Microscopically the brittle fractures are called as cleavage facets and ductile fractures are called as dimples (Ozden et al. 2007).





Full Scale 1632 cts Cursor: 0.000 keV Fig.7(a). SEM and EDX analysis of fractured tensile strength for sample A0

Figure 7 (a and b) shows the SEM and EDX analysis images for fracture tensile specimens for sample A0 and A3 respectively. It is observed from the images that the specimens have undergone both brittle and ductile fractures. Resisting initial cracks and its growth in the matrix are the major factors in achieving the maximum tensile strength. Aluminium 7075 is a ductile material and more dimple structure is observed from the Figure 7 (a) for sample A0. CDA being a soft material increases the elastic nature of aluminium alloy and provides slow propagation of cracks.

Addition of reinforcements in aluminium 7075 may leads to more cleavage facets causing brittle failure in the matrix. The presence of hard ceramic particles in the matrix resists the applied load during plastic deformation. The thermal mismatch between reinforced particles and aluminium matrix may also be the reason for increase in resistance. The thermal mismatch occurs in the composite materials since the coefficient of thermal expansion is high for aluminium matrix and is low for the reinforced particles. The increase in percentage of B_4C reduces the elastic

deformation in the composite. Figure 7 (b) signify clustering and debonding of reinforcements in the matrix alloy causes voids and micro cracks in the composites. The cracking of particles and brittleness along the surface of composite material is also clear.

The major elements present in Al 7075 has given in Table 1 are Al, Zn, Cu etc and the elements can be identified from Figure 7 (a) for sample A0. The major content in CDA particles is Si from the XRF analysis given in Table 2. From EDX figure 7 (b) for sample A3 it can be identified that the Si content is more and available in the fabricated composite material. The presence of Si confirms and proves that the reinforcements are present in the tensile fractured surfaces.



Full Scale 1632 cts Cursor: 0.000 keV Fig.7(b). SEM and EDX analysis of fractured tensile strength for sample A3

Evaluation of Wear behaviour

The graphical representation of wear rate and coefficient of friction is shown in Figure 8 and 9 respectively. Wear and coefficient of friction were studied to identify the materials resistance against sliding with other surfaces. Wear rate reduces due to less plastic deformation between the sliding contact area and the sliding material. Less plastic deformation is achieved due to the presence of reinforcements which minimizes the shear stress transfer during sliding. During sliding action oxidation of metallic particles occurs and a layer is formed on the pin surface. The layer formed undergoes distortion, spalling and fracture during sliding action. This layer formed between the mating surface offers resistance due to the dilution of metallic contact of the surface.

The wear rate values were found to be 0.005016, 0.003541, 0.002851 and 0.002027 and coefficient of friction was found to be 0.394, 0.357, 0.341 and 0.310 for A0, A1, A2 and A3 samples respectively. The wear rate gradually decreases for all composite samples when compared with the base alloy. The lowest wear rate value was found for sample A3 having 7.5% $B_4C - 2.5\%$ CDA composition of reinforcements and 59.58% of increased resistance was achieved when compared with the fabricated aluminium 7075 alloy.



Fig.8. Effects of B₄C and CDA on Wear



Fig.9. Effects of B_4C and CDA on coefficient of friction

When the pin surface slides over the abrasive disc, material removal occurs due to microscopic removal mechanisms in sliding or rolling contact. Abrasive wear is caused by micro-cutting and microploughing. From the literature reports of Baradeswaran et al. (2013); Manikandan, R et al. (2020); Urena et al. (2009); Maleque et al.(2017) when wear is low, deformation occurs by micro-ploughing and at high wear, deformation occurs by micro-cutting mechanisms. The wear resistance is governed by transition from micro-ploughing to micro-cutting mechanisms for composite materials against sliding force.

The SEM with EDX images for the worn out surface are shown in Figure 10 (a and b) for sample A0 and A3 respectively. It has been observed that microploughing and micro-cutting wear mechanism have occurred in the fabricated samples. From the worn surface it is clear that more micro cuttings can be seen which leads to high wear rate in the samples. High wear rate is observed in sample A0 where no reinforcements are added and the other reinforced samples.





Fig.10(a). SEM and EDX analysis of worn surface for sample A0





Fig.10(b). SEM and EDX analysis of worn surface for sample A3

The wear resistance is governed by transition from micro-ploughing to micro-cutting mechanisms

for composite materials against sliding force. The conversion from micro-ploughing to micro-cutting is comparatively less for specimen A3 when compared with samples A0 and it has been concluded that at 7.5% B₄C - 2.5% CDA of reinforcement wear resistance has increased. The EDX analysis shows the peaks of aluminium and reinforced particles along with Fe and O elements. The mechanically mixed layer formed between the pin and the surface plays a vital role in reduction of wear in the composite samples. The oxide and iron peaks present in sample A3 is more when compared with samples A0 which confirms that the wear resistance has increased.

CONCLUSIONS

In the current study the effects of reinforcing B_4C and CDA into Al (Al 7075) were carried and the following conclusions are summarised below:

- Fabrications of composite samples were done by stir casting technique.
- Optical microstructure images reveal comparatively uniform dispersion of B₄C and CDA reinforcements in aluminium matrix.
- Hardness has gradually increased in addition of reinforcements and a maximum of 30.90% increment in hardness for sample A3 composites was found to that of base alloy.
- The tensile strength has also gradually increased in addition of reinforcements and a maximum of 56% increment in ultimate tensile strength was achieved for sample A3 hybrid composites to that of aluminium alloy.
- Wear rate has gradually decreased for all the hybrid composites and a maximum of 59.58% increase in wear resistance for sample A3 was attained to that of aluminium alloy.
- Dimples, transgranular cleavages and micro ploughing, micro cutting are seen in the fractured and worn specimens respectively.

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