International Journal of Engineering Trends and Technology
ISSN: 2231 – 5381 /doi:10.14445/22315381/IJETT-V69I7P234

Volume 69 Issue 7, 270-277, July, 2021

Processing, Characterization and Fractography of Dual Step Stir Casted Al6061/(Si_3N_4+SiC) Composites

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Abstract *- The present research article discusses fabrication, micro structural investigation and mechanical behavior of Al6061/(Si3N4+SiC) composite. High strength at the cost of toughness is very often in metallic composites reinforced with hard ceramic particles. Aerospace, automobile and marine industries demand tough and high strength materials. Simultaneous improvement in strength and fracture toughness is the major concern of the current study. To attain the objective, two reinforcement particles of dissimilar shape and sizes were chosen. Small quantity of micron sized SiC particles and elongated rodlike submicron Si3N4 particles were utilized to reinforce the Al6061 alloy matrix. Aforementioned combination of reinforcement and aluminium matrix is unique in itself. Three different compositions including base alloy were fabricated by two step stir casting method for analysis. For micro structural characterization, an optical microscope and FESEM (field emission scanning electron microscope) with EDS were used. Fractured surface of the tensile test was also investigated by FESEM.*

Keywords — *Elongated rodlike submicron Si_3N_4; Al6061/(Si_3N_4+SiC) composite; two step stir casting; FESEM; fractured surface analysis*.

I. INTRODUCTION

Metal matrix composites (MMCs) have been intensively researched over the past few years and many new high strength to weight materials have been developed. Most of these materials have been developed for their use in aeronautics, space industries but some are being used in automotive, transportation and biomedical applications too. In general, according to reinforcement, the three main types of MMCs are continuous fiber, discontinuous fiber (or whiskers) and particulate reinforced metal matrix composites [1-2]. Particle reinforced metal matrix composites are more extensively used because of their economical cost, easy availability, isotropic properties and their ease to process by simple conventional techniques [3]. Carbides (SiC, B_4C, TiC etc.), nitrides (Si_3N_4, AlN), oxides (Al_2O_3, TiO_2) and borides (TiB_2) are most widely used to reinforce the commonly used metal matrix materials like Mg, Al, Ti [4-7]. Among these metal matrix, aluminum based MMCs have wide range of industrial applications [2, 8, 9]. In recent days, particulate hybrid aluminum matrix composites (PHAMCs) are becoming very popular. It contains two or more than two different types of reinforcement particles [10]. PHAMCs have the potential to meet the demands of advanced engineering applications [11].

Researchers always got attracted towards a thought of creating economical and superior material. In light to this motivational frame of mind, two different Al6061/(Si3N4+SiC)/(wt%X+wt%Y)[1]$_p$ composites were produced in the present exploration. SiC and Si3N4 particles are very popular reinforcements that have been used frequently in metal matrix for decades as evident from past research articles [6, 7, 12-19]. However, they are rarely reported to be used as together. So it was decided to conduct the study using them in combination to reinforce PHAMCs for an exceptional composition.

Al6061 alloy contains Mg and Si as major constituent.. Its strength basically depends on proper processing of Mg2Si precipitates in aluminum [20]. It is a light weight, cost effective alloy that possess excellent machining and casting abilities with reasonable mechanical properties. It also has good resistance to corrosion [21]. Besides aforesaid characteristics, Al6061 has some drawbacks, that it contains rough particles and needle/plate like eutectic silicon which depreciate mechanical properties [20]. Silicon nitride (Si_3N_4) & silicon carbide (SiC) are excellent materials for high temperature applications. They are having unique combination of properties like low density, high hardness, better wear and corrosion resistance [22-24]. Silicon nitride is chemically stable and has excellent ability to retain mechanical strength at elevated temperature, it is extremely hard and comparatively tough as well. It is

[1] Percentage by weight for Si_3N_4 and SiC particles respectively

Element	Mn	Fe	Mg	Si	Cu	Zn	Ti	Cr	Other	Al
Composition	0.10	0.70	0.80	0.60	0.40	0.10	0.05	0.05	Below 0.15	Balance

evident from the literature that elongated grain of silicon nitride improves its fracture strength [25-26]. It was also reported that dispersion of SiC whiskers in Si_3N_4 matrix improves the toughness of ceramic composite [27]. Compared to nano sized SiC particle, micron sized SiC particle have better modifying effect on dendrites to reduce dendrite length [28].

'Stir casting' is a class of liquid state processing technique, supported by various literatures, which found it to be economical, simple, flexible, having high material yield, suitable for mass production and give better results [2,29]. Proper selection of stirring process parameters is quite necessary for getting anticipated properties of composite mterials since the distribution of reinforcement regulated by these stirring parameters. Wettability of reinforcement is one of the common problems associated with this method [30]. A strong interface between reinforcement and matrix critically depends upon wettability level of reinforcement by liquid matrix metal [31,32]. Ability of the molten metal to spread over the surface of reinforcement particle is termed as wettability [33]. Weak wettability is responsible for poor load transfer between the matrix and reinforcement, agglomeration, porosity around the particle (probably due to solidification shrinkage) which leads to inferior mechanical, wear and corrosion characteristics of the composite [34,35]. Numerous different techniques by several researchers have so far been used to improve the wettability. Efforts made mainly includes (1) Addition of alloying element/wetting agent for example addition of magnesium {<1wt%}which reduces surface tension of the melt and further reduces solid-liquid interface energy by aiding chemical reaction; (2) Ceramic particle e.g. Ni, Cu and Co coating; (3) Preheating of reinforced particle; (4) Performing stir casting in steps; (5) Ultrasonic assisted vibrations etc. [15,35,36].

In current research, to avoid wettability problem, three major techniques were opted that are addition of alloying element, reinforcements preheating and performing stir casting in two steps.

II. MATERIALS AND METHOD

A. Materials

All the raw materials were purchased from M/S Parashawamani Metals Mumbai, India. Al6061 alloy was utilized as matrix while with more than 99% purity, SiC and Si_3N_4 particles were chosen as reinforcements for composite fabrication. A brief description about chemical composition and properties of the materials are listed in table 1 and 2. Figure1 (a), and (b) shows the FESEM image of Si_3N_4 and SiC powder respectively.

TABLE 1.
CHEMICAL COMPOSITION OF Al6061 ALLOY.

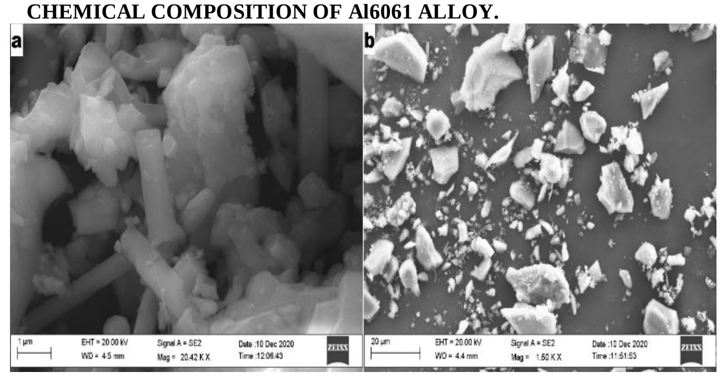

Figure 1. FESEM image of received (a) Silicon nitride powder, and (b) Silicon carbide powder

B. Methods

a) Processing of composite

Total three compositions including base alloy were prepared by dual step stir casting. For fabrication of composites, electric furnace was used. Small pieces of Al6061 rods were charged into the temperature controlled furnace and melting was allowed until the complete molten state with a uniform temperature of 750℃ was achieved. The time taken to attain the state was 1.5 hours. After that, melt was allowed to cool to 625℃ in the furnace to reach it to a semi solid state. 0.2wt% magnesium and coverall were added, to increase the wettability and to remove the impurities & gases. After scooping out the impurities floated over the top of the crucible, manual stirring is done and when a proper vortex is formed a fine mixture of seperately preheated reinforcement (at 550℃) was introduced into the semi solid melt. Manual stirring of the slurry was performed for about 10 minutes.

As the manual stirring completed, the composite slurry was started to reheat and maintain a temperature of 750℃±30℃ . A second stirring was then performed using a three bladed well designed, well positioned, speed regulated mechanical stirrer at 550 rpm for 10 minutes . To avoid the spattering of the melt, impeller of the stirrer is preheated and a graphite coating was done before the insertion to avoid any reaction with impeller's material. Now, the metal matrix composite poured into preheated die and then allowed to solidify. Finally the casts were machined to prepare samples according to ASTM standards for different tests. The utilized compositions to prepare the composites have been shown in table 3.

TABLE 2. PROPERTIES OF ALLOY AND CERAMIC PARTICLES.

Material	Form	Density(g/cc)	Melting Temp. (℃)	Reinforcement Size (μm)	CTE (10^{-6}/℃)	Hardness (GPa)	Young's modulus (GPa)
Al6061	Rod	2.70	650	N/A*	23.4	0.9	70
Si_3N_4	Powder	3.25	1900	1-5	3[18]	20	310
SiC	Powder	3.17	2730	12-18	4[5]	28	460[5]

* Not applicable

TABLE 3. COMPOSITIONAL DETAILS OF COMPOSITE

Sample ID	Sample-Composition ($wt\%_X+wt\%_Y$)	Al (g)	Si_3N_4 (g)	SiC (g)
S0	Al6061/(0.0% Si_3N_4+0.0%SiC)	1600	0	0
S1	Al6061/(0.5%Si_3N_4+0.5%SiC)	1600	8	8
S2	Al6061/(0.25%Si_3N_4+0.75%SiC)	1600	4	8

b) Density, hardness and material characterization

The prepared samples were investigated to determine composite density, hardness, microstructure, tensile strength and ductility. Experimental density (actual density) measurement was performed at room temperature (25°C) by Archimedes principle while theoretical density was obtained by the rule of mixture. After finding thease two values, porosity level in terms of percentage was checked. To weigh the samples, a high precision digital electronic weighing machine with an accuracy of 0.01 mg was used.

Rule of mixture:

For density measurement:

$$\rho_{Al6061/SiC/Si3N4} = wt\%\ Al6061 \times \rho_{Al6061} + wt\%\ Si3N4 \times \rho_{Si3N4} + wt\%\ SiC \times \rho_{SiC} \quad (1)$$

Percentage porosity is given by:

$$\%\ porosity = \{(\rho_{theoretical} - \rho_{actual}) \div \rho_{theoretical}\} \times 100\ \% \quad (2)$$

Where ρ= density; wt% = weight percentage

Hardness of Al6061 alloy and prepared composite was measured by Vickers microhardness tester. A square based pyramidal shape diamond indenter, 1000gf test load and 15s dwell time was used for the microhardness study. ASTM: E384-08 standard was followed to conduct the experiment. Average of five readings, were taken as hardness of the sample.

For micro structural studies, optical and SEM micrographs were taken. All the standard considerations were followed for sample preparation. Microstructure of alloy and composite were examined by "Carl-Zeiss Axio Scope 5 optical microscope". Morphology of particles, micro structural investigations and fractographic analysis of tensile test fractured samples were observed by "Carl Zeiss make Ultra Plus field emission scanning electron microscope" (FESEM).

Tensile test was performed on "Mechatronic Control systems", India make Mech.CS.UTE 40T UTM. ASTM: E8-95 standard was followed to prepare the test specimen (as in figure 2). Cross head speed was set at 1.4mm/min during the test.

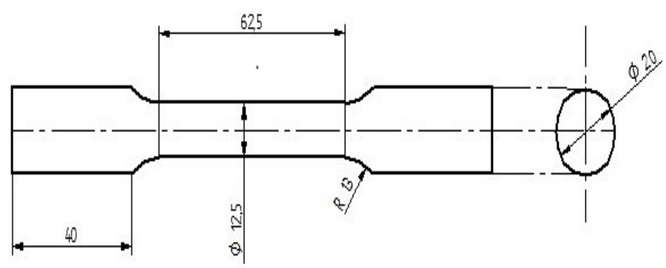

Figure 2. Draft of tensile test specimen (all the dimensions in millimeter).

III. RESULTS AND DISCUSSION

A. Density and porosity

TABLE 2. DENSITY AND POROSITY VARIATION FOR ALL THE THREE COMPOSITIONS.

Sample ID	Theoretical density (g/cc)	Actual density (g/cc)	%porosity	% Density rise (in compare to S0)
S0	2.7000	2.663	1.37	0
S1	2.7051	2.667	1.41	0.15
S2	2.7049	2.665	1.48	0.08

Theoretical and actual density with percentage porosity and density rise for different samples (sample IDs S0,S1,S2) are tabulated in table 4. It is found that the porosity is within the acceptable limit. Low porosity is a good sign to rely on stir casting for the production of composite. Maximum porosity was found in sample S2 which is 1.48%. Highest increment in density was found in S1 (i.e. 0.15%). The rise in density is certainly due to high density reinforcement particles.

B. Microhardness

Vickers microhardness testing results are illustrated in the figure 3. Five readings for each samples were recorded and it was observed that, all the samples were having very small differences in all five readings which were taken at different places as per the standard norms. This uniformity in the hardness is an indication of uniform distribution of hard ceramic particles. Hardness of both composite was significantly higher than that of base alloy. Grain refinement due to the presence of reinforcement and high dislocation densities due to the reinfocement presence and mismatch in coefficient of thermal expansion (CTE) are the genuine cause behind the rise in hardness[11,16]. Highest hardness was observed for sample S2 since it contains harder reinforcement (SiC) in little large amount comparing to sample S1.

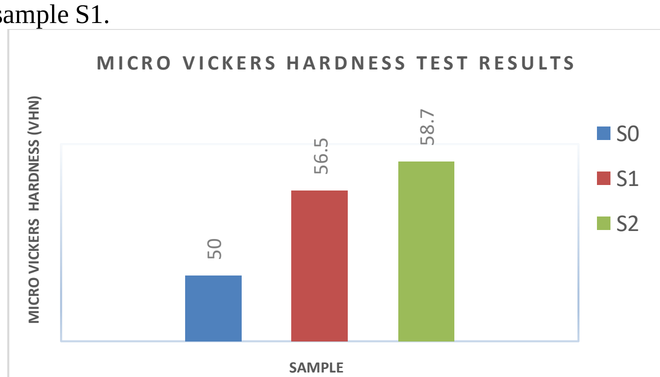

Figure 3. Vickers microhardness test result, illustrates that Al6061/(0.5%Si_3N_4+0.5%SiC) composite has highest hardness.

C. Microstructure

Micro structural studies are quite useful for prediction of properties of end material. Figure 4 (a), (b) and (c) represent optical micrographs of sample S0, S1 and S2 respectively. Figure 4 (a) reveals the coarse and fine grained α- Al dendrites with the micro segregation of solute atoms like Mg, Si, Fe and Cu at the interdendritic regions [37]. It is evident from the past researches that the severity of micro segregation depends upon the growth rate while formation of eutectic phases at interdendritic region governed by Mg and Si, present in Al6061 [37, 38]. In optical micrographs as in figure 4 (b) and (c), grain sizes of aluminum getting reduced, consequently the grain boundary concentration increasing. Higher the boundary concentration, higher will be the obstacle for dislocation movement which in turns, improved mechanical and tribological characteristics of the composites. For details of microstructural investigation, Field Emission Scanning Electron Microscope (FESEM) with EDS was used. FESEM, micrographs for base alloy and composites are shown in figure 5 (a), (b) and (c). A clear cut grain refinement is showing in composite samples, composite's micrographs have small grain boundaries with uniform distribution of reinforcement particles without significant particle clustering and porosity in the matrix. Dense microstructure of the composite indicates improved strength, stiffness, dimensional stability and enhanced life of the component. Energy dispersive spectroscopy (EDS) analysis (figure 6) was carried out to confirm the presence of silicon carbide and silicon nitride particles in aluminum matririx. Optimized processing parameters are responsible for the desired microstructure. A reaction free interface between constituting phases was observed from FESEM image as shown in figure 7.

D. Mechanical Properties

The fabricated composites of present research reveal higher tensile strength along with higher impact strength and more ductility in comparison to base alloy. Such kind of

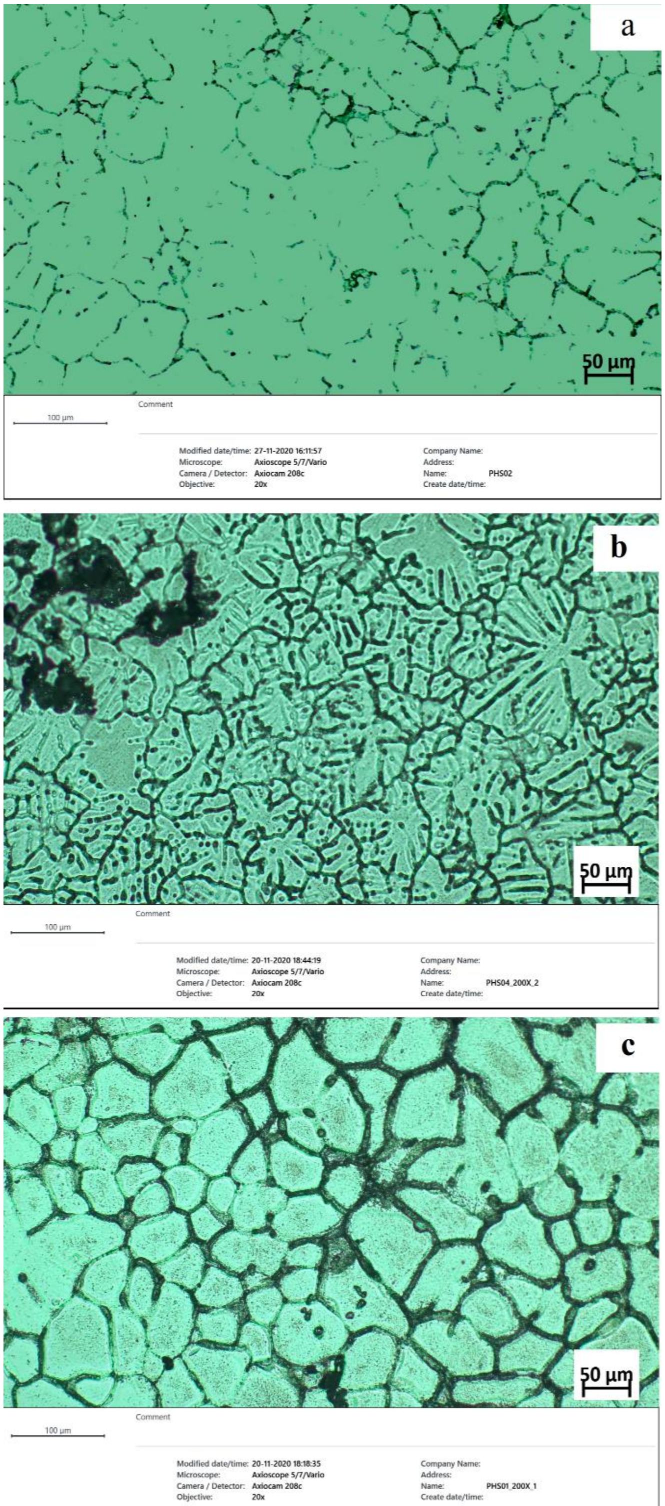

Figure 4. Optical micrograph of (a) Al6061 alloy, (b) Al6061/(0.5%Si_3N_4+0.5%SiC) composite, (c) Al6061/ (0.25%Si_3N_4+ 0.75%SiC) composite, (b) and (c) illustrate the uniform dispersion of reinforcements

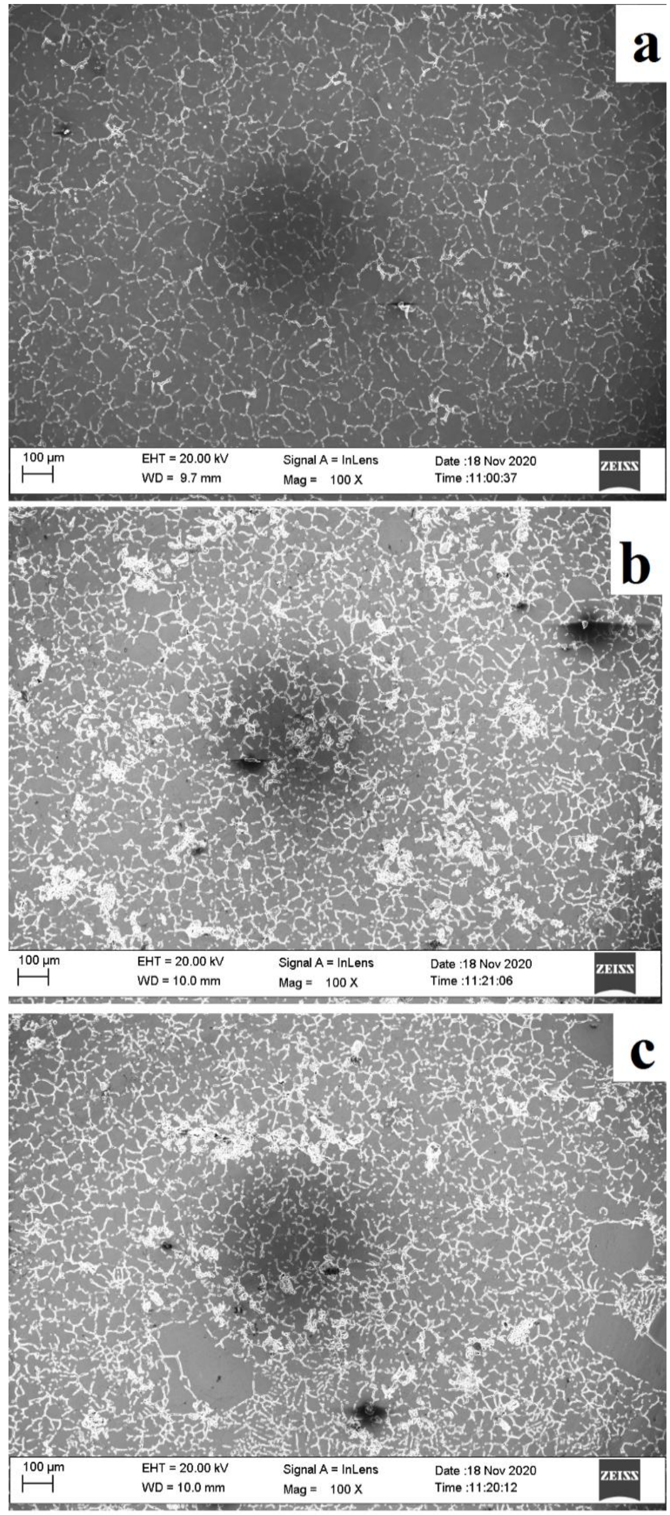

Figure 5. FESEM micrographs of (a) as cast Al6061 alloy, (b) Al6061/(0.5%Si_3N_4+0.5%SiC) composite, and (c) Al6061/ (0.25%Si_3N_4+ 0.75%SiC), represent the proof of sound casting.

Simultaneous improvement in properties is rarely found in the results of different researches. Variation of ultimate tensile strength (UTS), impact strength and % elongation for different compositions are represented in figure 8 (a), (b) and (c) respectively.

Grain refinement with direct and indirect strengthening is the usual cause of improved tensile strength [26] while, elongated rodlike structure of Si_3N_4, that surrounds the SiC particles provide a flexible support and give extra strength to SiC particles. A represnative diagram as in figure 9 is showing the interaction between SiC and rodlike Si_3N_4 particles within the matrix. It has been proved in the past researches, that use of continuous fibers, whiskers and platelet ceramic reinforcements may improve the toughness. Elongated rodlike Si_3N_4 particles give a buckling effect which results in a small scale damping. It is the main cause behind the unusual combination of property rise i.e. high tensile strength with improved fracture toughness and improved ductility. SiC and Si_3N_4 altogether hinder the dislocation movement and improves overall strength of composite. Presence of thin amorphous inter granular film in $Si3N_4$ [39] may also be an additional reason for fracture toughness improvement. Improved fracture toughness is the cause of higher % elongation.

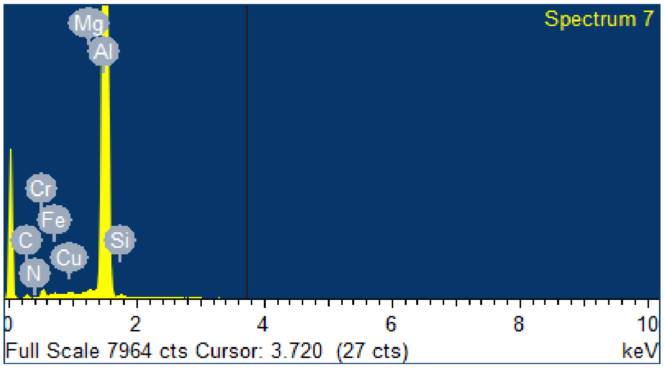

Figure 6. EDS analysis of Al6061/(Si3N4+SiC) composite.

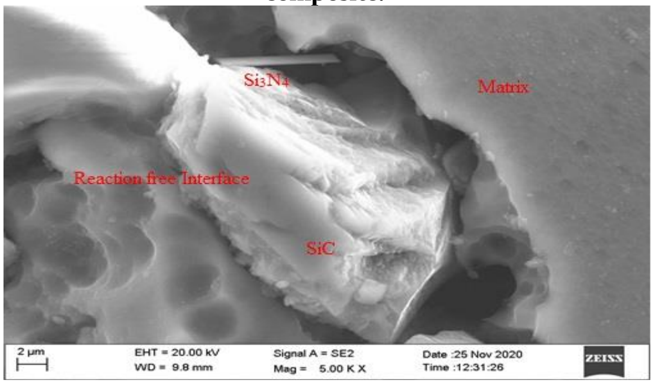

Figure 7. FESEM photograph of Al6061/(Si3N4+SiC) composite illustrating excellent bond between matrix alloy and reinforcements in absence of interfacial reaction.

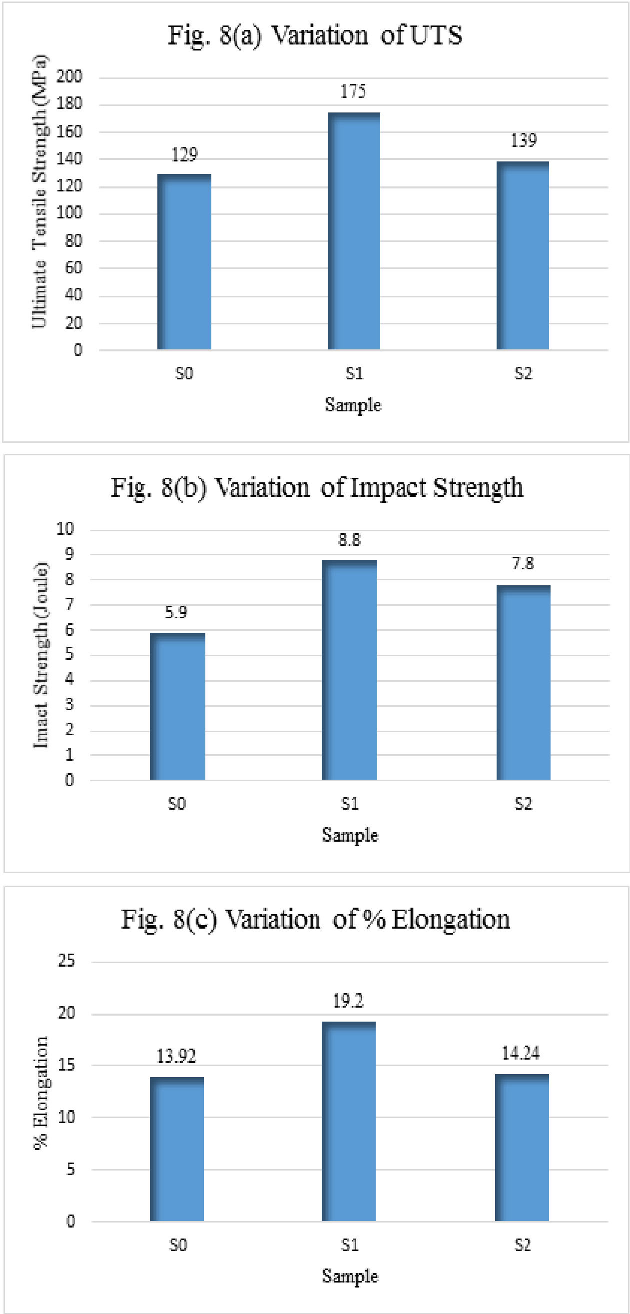

Figure 8. a-c. . Comparision among all composition samples on the basis of ultimate tensile strength, impact strength and % elongation.

E. Fractured Surface Analysis of Tensile Test Samples

FESEM images of fractured surfaces of S0, S1 and S2 samples are being represented by Figure 10 (a), (b) and (c) respectively. All images contain small facets and tear ridges, which is the indication of ductile fracture [40]. Fractured surface of S1 composite, which are having number of shallow voids/dimples with flat and smooth

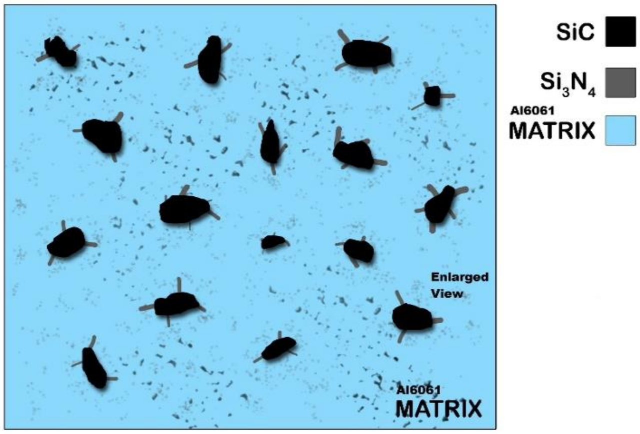

Figure 9. Represntative diagram, showing interaction between SiC and rodlike Si_3N_4 particle in Al061 matrix (key concept of the study).

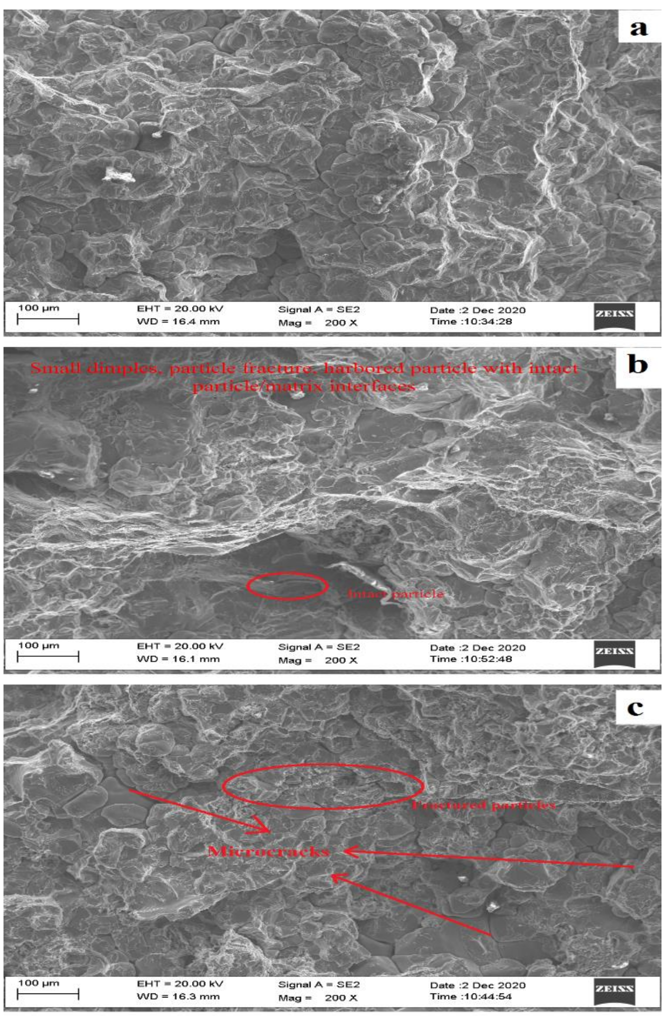

Figure 10. FESEM fractograph of (a) Al6061 alloy [S0], (b) Al6061/(0.5%Si_3N_4+0.5%SiC) composite [S1], and (c) Al6061/(0.25%Si_3N_4+0.75%SiC) composite [S2].

mirror regions, whereas S2 composite possess flat regions with microcracks. These Small microcracks indicate to brittle fracture. Composite surfaces does not show any significant evidence of particle debonding, which is the indication of good interfacial bonding between constituting phases. Intact particle- matrix interface represents that, the shear strength at the interface is quite higher than the particle fracture strength. It is also found that the particles are well harbored in the matrix [3]. On the basis of fractured surface analysis it has been expected to have better mechanical properties with the composites.

IV. CONCLUSIONS

1. Elongated, rod like structure of Si3N4, which surrounds the SiC particles provides a flexible support and give extra strength to SiC particles and matrix. It is the main cause behind the unusual combination of property rise i.e. high strength with improved fracture toughness and ductility. Elongated Si3N4 particles give a buckling effect which results in a small scale damping and finally in this way improves the fracture toughness. SiC and Si3N4 altogether hinder the dislocation movement and improves overall strength of composite.
2. Homogeneous distribution of reinforcement particles is an evidence of optimized processing paramateres. Optimization of processing parameters plays a key role for effectiveness of two step stir casting method while homogeneous distribution of reinforcement improves mechanical, physical and tribological properties of the composite.
3. Both composites have better mechanical properties, as compared to the base alloy. Al6061/(0.5%Si3N4+0.5%SiC) composite (S1) possess better properties as compared to Al6061/(0.25%Si3N4+0.5%SiC) composite (S2). Morphology of Si3N4 and its combination with SiC particles in equal proportion are the governing cause for better properties of S1 rather than S2. Equal proportion of the reinforcements provides better properties so it is concluded that proportion of reinforcing phases play a quite important role in betterment of final composite material.

FUNDING

This research received no specific grant from any funding agencies in the public, commercial, or not-for-profit sectors.

DECLARATION OF CONFLICTING INTERESTS

The authors declare that there is no conflict of interest.

ACKNOWLEDGMENTS

The authors would like to acknowledge their sincere thanks to department of Material Science and Metallurgical Engineering (MSME), Maulana Azad National Institute of Technology (MANIT) Bhopal, India and Central Instrumentation Facility (CIF) at Indian Institute of Science Education and Research (IISER), Bhopal, India, for accessing their testing facilities. The authors also express their deep sense of gratitude to Dr. C. Sasikumar, Assistant Professor, MSME department, MANIT, Bhopal for his continued support and cooperation.

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