Development of Al-SiC composite material from rice husk and its parametric assessment

Madhusudan Baghel1, C M Krishna2 and S Suresh3
1 Department of Mechanical Engineering, Maulana Azad National Institute of Technology, Bhopal-462003, MP, India
2 Department of Chemical Engineering, Maulana Azad National Institute of Technology, Bhopal-462003, MP, India
E-mail: baghelmadhusudan@gmail.com
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Abstract
In this research work, the development of Al-SiC composite material from rice husk and its parametric assessment is done using a CNC milling machine. They are further surface characterized, and mechanical properties such as BET surface area, SEM-EDX, and XRD, fracture toughness, tensile, and bending strength are studied. The machinability of the components is investigated for selected values of input-output parameters. Three castings, each with different particulate reinforcement combinations, are made with aluminum alloy (6061) using the stir casting method. BET surface area of extracted silica and Al-SiC composite material was found 374 m² g⁻¹ and 150 m² g⁻¹, respectively. From results of BET surface area revealed that silica obtained from rice husk is more heterogeneous with a large surface area. A heterogeneous surface with larger pores was found through SEM images. XRD diffraction peaks show changes of amorphous silica into crystallinity in the composite material. The results also indicate that fracture toughness is very good at low temperatures and good machinability on CNC milling machines makes it suitable for aerospace applications.

1. Introduction

Metal matrix composites are finding a prominent place in many industries instead of corresponding metals for a variety of applications as they offer good mechanical properties. Metal matrix composites (MMCs) made of aluminum alloy as a base are very widely used in some applications like automobile and aerospace sector [1, 2]. Of late, Aluminum silicon carbide (Al-SiC), one of the Aluminum-based MMCs with superior properties, such as fracture toughness and good wear resistance, found its application in the aerospace industry. However, the machinability of Al-SiC composites is a challenge when making dies on electric discharge machines (EDMs) due to the microparticle size of silicon carbide. While machining on EDM, silicon being non-conductive of electricity and high melting point, remains in particle form, whereas aluminum melts when the arc is discharged. Hence it is important to develop silicon carbide in nano form to make it suitable for non-conventional machining such as EDM.

There are some cheaper resources available for reinforcement with aluminum, such as agricultural wastes and industrial wastes. As these can be recycled and the products can be reinforced with aluminum, they have gained popularity over recent years [3, 4]. Fly ash (waste by-product of power plant) is one of the cheaper reinforcements used for low-cost aluminum composites. Selvam et al (2013) fabricated AA6061 composites reinforced with 0, 4, 8, and 12 wt% fly ash using compocasting and observed their microstructure and mechanical properties. Ultimate tensile strength and microhardness of 12 wt% fly ash/AA6061 composites were improved by 56.95% and 132.21%, respectively, compared to AA6061 alloy [5]. Size of fly ash particles also affects the properties of composites and it was found that narrow size range of fly ash particles reinforced aluminum composites showed better properties than wider size range particles [6]. Many researchers also used bamboo leaf ash (agro waste) as reinforcement in aluminum matrix composites. B. Kumar et al (2017) observed the effect of various amount of bamboo leaf ash on tensile strength and hardness of composites prepared via stir casting. Maximum value of hardness (99.3 BHN) and tensile strength (177.3 MPa) were obtained in case of...
4 wt% bamboo leaf ash [7]. Al-4.5Cu was reinforced with bamboo leaf ash by Rao et al. (2020) and corrosion behaviour was studied before and after laser surface melting (LSM). Hardness and distribution of reinforcement were improved with LSM [8]. Coconut shell ash is another agricultural waste that can be used as reinforcement to produce low-cost composites. Kumar et al. (2018) used stir casting to fabricate ZrO2 and coconut shell ash reinforced Al6082 composites and observed the effect of reinforcements on mechanical properties. Addition of coconut shell ash improved the flexural strength and reduced the impact strength [9]. Taguchi Taguchi L9 orthogonal array and genetic algorithm were used by M. Arulraj et al. (2018) for optimization of parameters during impact testing on LM24-SiC-coconut shell ash composites. Composites showed 20% improvement in impact strength at optimum value [10]. Bagasse ash (waste product of sugar industry) has been used as reinforcement in Al matrix composites by many researchers. S. Subramanian et al. (2019) fabricated Al-Si10-Mg reinforced with bagasse ash using stir casting. Wear test was conducted at load of 10, 20, and 30 N with constant sliding distance and speed. 9 wt% bagasse ash reinforced Al composites showed lowest coefficient of friction and wear rate [11]. Mechanical properties of bagasse ash reinforced Al composites were evaluated by Shankar et al. (2018). Impact strength and hardness were improved by 15.5% and 33.4%, respectively, with addition of 1.2 wt% of bagasse ash [12]. Hardness and tribological properties of egg shell powder reinforced Al7075 composites were discussed by A. Kumar et al. (2021), reinforced via stir casting. Egg shell powder was in the form of calcium oxide (hard material) resulting in maximum value of hardness (197 HV) [13]. Carbonized and uncarbonized egg shell were used as reinforcement in Al-Cu-Mg composites by Hassan et al. (2013) using stir casting. Better mechanical and physical properties were observed in case of carbonized egg shell reinforced composites [14]. There are some other cheap resources that can be used as reinforcement in Al matrix composites such as palm kernel shell [15], red mud [16, 17], silica sand [18], lemon grass ash [19], groundnut shell ash [20], etc.

In the current study, silicon carbide is extracted from rice husk in nano form by chemical synthesis process as it is widely used as reinforcement material because of its affordability, broad applicability, and high modulus [21]. Numerous processing techniques are being used to develop MMCs, and in this work, stir casting is adopted for producing Al-SiC MMC. Characteristics of the resultant composite improve with the addition of silicon due to matrix strength through the solid solution at reinforcement/matrix interfaces. Researchers used various methods for extraction of SiC from rice husk, fly ash, marble sludge powder, etc. Alane and Adewale investigated the impact of SiC and rice husk ash reinforcements on mechanical characteristics of Mg-SiC/Al hybrid composite. They concluded that porosity values are less than 2.5% in all cases, and thus, the resultant castings are of good quality [22]. Mishra et al. (2013) used filler material, marble sludge powder, to reduce total void content in concrete [23]. The blended elements developed the highest strength, increasing as the marble content increased. Parmoda et al. (2013) studied the properties of composites of graphene that contain surface oxygen functionalities with metal halide vapors and then exposed to water vapor [4]. Sharma et al. (2012) studied wear characteristics of sisal fiber composite. They observed that the wear increases as the fiber content increases up to some extent and then decreases [24]. They obtained 60% addition of fiber resulted in minimum wear value. Suresh et al. (2012) prepared impregnated activated materials from cashew nut shells for adsorption of benzene vapor [5].

Many researchers have studied the mechanical characteristics of MMCs. Rajesh et al. [25] used stir casting to prepare a metal matrix composite of Al7075 and SiC. They carried out mechanical characterization and concluded that the hardness of composites was proportionately influenced by percentage of SiC. Manivannan et al. [26] used the stir casting method based on ultrasonic cavitation to fabricate Al6061 reinforced with nano-sized SiC. A nanocomposite with SiC particles of 1.2% yielded maximum ultimate tensile strength. Saheb [27] developed aluminum-based graphite particulate MMCs and silicon carbide particulate MMCs. They found maximum hardness in the case of SiC particles of 25% and graphite particles of 4% weight fractions. Reddy [28] considered 7072/SiCp for studying their fracture and tensile behavior after casting MMCs using gravity die casting and found that maximum tensile strength was obtained for addition of 10% SiC. Ekiçi et al. [29] found the influences of volume fraction, dispersion, size, and concentration of SiC particles on residual stress, strain distribution, and indentation depth of an Al 1080/SiC particles reinforcement MMCs. Sakhthivel et al. [30] examined the stir casting method and developed the 2618 aluminum alloy MMCs reinforced with SiC particles at two levels of size up to 10 weight % SiC and subsequent forging operation. Kalkani and Yilmaz [31] studied the characteristics of MMC of 7075 Al-SiCp in the range of weight % of SiC from 10 to 30% using vertical pressure squeeze casting. They obtained an increase in hardness from 133 to 188 VHC for an increase of SiC from 10 to 30%.

The fracture toughness characteristic of MMCs is very important for application in the aerospace industry. Park et al. [32] investigated fracture toughness and behavior of 6061 Al-Mn Si alloy with 20% volume Al2O3 based polycrystalline ceramic microspheres. They concluded that the addition of particulate matrix decreases fracture toughness. Rabiei et al. [33] compared predicted fracture toughness with experimental fracture toughness. It was observed that ductile fraction of ligament in between the reinforcements was responsible for failure of MMCs. Effect of reinforcement (Al2O3) density on fracture behavior of Al2O3/Al6061 composites was
evaluated by Hung et al [34]. Their studies showed that surface preparation affects fracture resistance. Mechanical properties of Al₂O₃-B₄C (fabricated via stir casting) were observed by Ramnath et al [35], and they found that the inclusion of boron carbide has a very good effect on fracture toughness. Ibrahim et al [36] concluded that microsized SiC (reinforcement) results in irregular distribution, agglomeration, and ineffectual wettability for squeeze or stir casting, powder metallurgy techniques, and spray forming. Sajjadi et al [37] designed a piece of single equipment for heat treatment of nanoparticles and also fabricated Al–Al₂O₃ micro as well as nano by 3 step mixing process, which includes heat treatment of nanoparticles. Results showed that the distribution of microparticles in Al melt is heterogeneous compared to nanoparticles. They found that the best distribution of particles occurred at a stirring speed of 300 rpm/min.

Researchers are interested in studying the optimization of machining characteristics in addition to mechanical characteristics as machining characteristics of MMCs are very important to study, especially when CNC machines are used for machining. Kollo et al (2010) studied machining nano SiC reinforced Al MMCs fabricated by planetary milling [38]. Tolouei-Rad and Bidhendi [39] maximized profit rate and estimated optimum value of machining parameters in milling operations using the feasible directions method. Surface roughness was predicted by Alauddin et al [40] using three dominant input parameters (feed, depth of cut, and cutting speed). Mechanical properties of SiC reinforced MMCs were affected by size of reinforcement particles (SiC) [41]. McDanielis [42] examined the effect of SiC whisker, module, or particulate reinforcement up to 40% on stress-strain behavior for various Al MMCs. Jit et al [43] fabricated MMCs of A384.1 and SiCₚ (particle size of 0.220 μm) containing different combinations of SiC in the range of 0 to 0.20% using modified stir casting technique. They observed that when SiC is added in Al alloy, it increases proof stress, but compressive strength increases until 0.15% addition and decreases further. Zhang et al [44] used a high-energy ultrasonic field to evaluate the microstructure of A356 alloy and their mechanical properties and observed the improvement in mechanical properties. M. Singla et al [45] developed Al-SiC composites using stir casting with varying wt% of SiC (5, 10, 15, 20, 25, and 30). 25 wt% SiC reinforced Al composites showed highest impact strength and hardness due to uniform distribution of reinforcement. Baradeswaran [46] examined stir casting method and fabricated a hybrid composite of Al7075 alloy reinforced with Al₂O₃-graphite. Results showed that presence of ceramic phase improved the tensile strength, hardness, flexural strength, and compressive strength. Maximum wear resistance was shown by hybrid composites containing graphite.

The current study is performed for the following three primary objectives: (1) Extraction of silicon carbide from rice husk by using chemical synthesis, (2) Preparation of Al-SiC composite using stir casting and performing different chemical and surface analysis tests and, (3) Evaluation of fracture toughness, tensile properties, and machinability of the resultant metal matrix composite in order to check the suitability for aerospace applications.

2. Materials and methods

2.1. Materials

AR grade chemicals were used for the experimental work. For dilution purposes, double distilled water (DDW) was used. Rice husk was taken from local farmers. Rice husk was manually cleaned initially for the synthesis process to eliminate the foreign particles. After that, actual synthesis was carried out using NaOH, which was procured from SRL Pvt. Ltd., Mumbai, and H₂SO₄ which was purchased from MOLYCHEM Pvt. Ltd. Pure Aluminum and rice husk were procured from the local market, and silicon carbide was extracted from rice husk. Coveral 11 (NaCl, KCl, Na₃SiF₆, NaAlF) is used as cleaning, drossing, and covering flex. Dry nitrogen gas is used as an antioxidant. Borax and magnesium are used to increase the wettability during fabrication.

2.2. Methods

2.2.1. Extraction of silica

Silica has been extracted from rice husk using dissolution followed by precipitation method. In this method, about 100 g of rice husk is weighed and washed with double distilled water and allowed to dry in an oven (Model: MOU-250, manufactured by Metrex Scientific Instruments Pvt. Ltd., New Delhi) for 24 h. The dried rice husk is burned in a muffle furnace (Model: MF 9M / 12M, manufactured by Metrex Scientific Instruments Pvt. Ltd., New Delhi) at 650°C for about 3 h with a sufficient quantity of air. The process is completed when pure white ash is formed. The ash is then mixed with 3N NaOH solution produced by dissolving 24 gms of NaOH in 200 ml distilled water. The solution and the ash are taken in a 1:7 ratio and heated in a round bottom flask with condenser water circulation. The clear solution formed is sodium silicate and is separated from residue by filtration. The solution is evaporated at 100°C for the removal of water. Add Conc. H₂SO₄ drop by drop to form silica and sodium sulfate. Sodium sulfate is soluble in water, and silica is filtered out and washed with water to remove traces of sodium sulfate [47]. The product has been dried in an oven overnight and kept for further use.
The various stages and chemical reactions during the process are given in figure 1. The chemical reactions involved are given in the equations (1) and (2).

$$\text{SiO}_2 + 2\text{NaOH} \rightarrow \text{Na}_2\text{SiO}_3$$  \hspace{1cm} (1)

$$\text{Na}_2\text{SiO}_3 + \text{H}_2\text{SO}_4 \rightarrow \text{SiO}_2 + \text{Na}_2\text{SO}_4 + \text{H}_2\text{O}$$  \hspace{1cm} (2)
For synthesis of SiC, obtained SiO₂ is placed in a graphite crucible covered with graphite powder. Crucible is then placed in an oven and heated at high temperature (1200 °C) followed by cooling to 80 °C. Then, obtained product is further heated at nearly 700 °C for 24 h to remove residual carbon. Chemical reaction involved is given in the equation (3).

\[
\text{SiO}_2 + 3\text{C} \rightarrow \text{SiC} + 2\text{CO}
\]  

(3)

2.2.2. Preparation of Al-SiC composite
Stir casting method (vortex) is used to prepare the composite. A resistance furnace is used to melt small pieces of aluminum in a crucible made of graphite at a temperature of 750 °C. Graphite paste is used for coating the stirring blade and die. At this stage, flux (Coverall 11) is added to molten aluminum for removal of dross, and it is followed by purging through dry Nitrogen gas (N₂) to avoid oxide formation. Wettability of SiC in the molten Al is improved by adding borax and magnesium (1%). The molten metal is then stirred at 300 rpm to form a vortex. At this stage, preheated SiC (900 °C for one hour) is added to the melt on the edge of the vortex for uniform mixing. The molten mixture of these two is stirred using the stirring mechanism of the stir casting arrangement for 15 min at 300 rev/min. The dross of the melt is gravity poured into the cast, and the specimens are removed after solidification.

2.2.3. Characterization of Al-SiC composites
SiC reinforced Al composites were characterized by different surface and chemical analyzers such as X-ray diffraction (XRD), scanning electron microscope (SEM) along with Brunauer–Emmett–Teller (BET), and

Figure 2. SEM image of (a) rice husk, (b) synthesized SiC, and (c) SiC reinforced Al composites.
energy-dispersive x-ray spectroscopy (EDX). Mechanical properties, viz, (i) fracture toughness, (ii) tensile, (iii) bending, and (iv) hardness strength was determined using universal testing machine, impact tester, etc.

3. Results and discussion

The BET surface area for Al-SiC surfaces and silica was 374 m² g⁻¹ and 150 m² g⁻¹, respectively. Figure 2 shows the particle shape, size, and surface morphology in the FE-SEM image for synthesized SiO₂ from rice husk. The average pore size was 402 and 323 micrometers, respectively, for silica and Al-SiC surfaces. This value shows silica obtained from rice husk was more heterogeneous with large surface area, indicating strong binding of particles in the fabricated composite material, Al-SiC composite. It also shows the presence of large particles of C as accumulation at some spot in the nanocatalyst sample. The chemical composition of extracted silica was 90% silica, and the rest of 10% was carbon and oxygen. Bulk density is found to be 80 to 160 Kg/m³. Distribution of SiC particles in Al matrix is represented in FESEM image as shown in figure 2 (c). homogeneous distribution of SiC particles can be observed due to preheating of SiC particles, adding Mg to improve wettability, and ultrasonic vibration to avoid the agglomeration. There is also no indication of crack formation due to using suitable process parameters during fabrication. Some small sized pores are also present in the casing due to air entrapment and improper flow of liquid metal during solidification.

3.1. X-ray diffraction (XRD)

XRD is a non-destructive technique that provides complete information about the chemical composition and crystallographic structure of natural and manufactured material.
The hkl values have been derived manually by solving the equations for different hkl. XRD of the silica extracted from rice husk and pure silica is represented in figures 3–4, confirming that significant particles are ZnO/SiC nanocatalysts. XRD results reveal strong C peaks and weak SiC peaks, confirming the presence of SiC.

3.2. Study of mechanical characteristics of Al-SiC composite

Prepared MMCs are subjected to various tests for studying different mechanical properties, such as fracture toughness, tensile strength, and bending strength. Also, its application was interpreted to aerospace parts. The testing has been carried out in Material Science and Metallurgical Engineering department at MANIT Bhopal, India.

The specimen is placed in the machine between the grips of UTM. An extensometer is used to record the change in gauge length during the test automatically. The change in length in the specimen is recorded by the machine while the load is applied, and the load is increased gradually till the rupture condition is achieved. 5 specimens were tested for tensile test and average was calculated. The plot of engineering stress and strain are shown in figure 5.

Figure 5. Engineering stress-strain curve.

Figure 6. Stress and strain analysis.
The results obtained from the test are analyzed to find elongation, yield strength, and tensile strength. From figure 6, the ultimate tensile stress is obtained as 157.42 ± 5.2 MPa, and the breaking stress recorded is 85.16 ± 2.6 MPa. The yield stress is 86.02 ± 3.4 MPa for 1 vol% SiC/Al composites. The improvement in tensile strength and breaking strength are 74.9% and 14%, respectively, compared to pure aluminum.

The statistical test method is widely used for measuring fracture toughness in the case of composite material. This method ensures sufficient conditions for plane strain conditions at the crack. The measured fracture toughness increases when test samples get thinner, providing a lower band under plane strain conditions. The dimensions of specimens used are 10 mm × 10 mm × 55 mm with 2 mm wide V-notch.

Orientation of fracture surface and the direction of crack growth play an important role in fracture toughness. Especially for composite materials, wherein fracture energies differ from plane to plane, there are significant differences in fracture toughness in different directions. Hence, the test specimen is pre-fatigued,

| Table 1. Fracture toughness for V-notch and plain specimen. |
|-----------------|-----------------|
|                 | V-notch | Plain specimen |
| σ in KPa        | 175     | 137            |
| a in m          | 0.004   | 0.00325        |
| Kc in KPa√m    | 19.6    | 43.5           |

Figure 7. (a) Fracture toughness test—V-notch, and (b) Fracture toughness test—Plain specimen.
specimen dimensions are measured, and a sample is loaded. Average of fracture toughness values of 5 samples for each condition (V-notch and plain specimen) was taken. The test results are shown in figure 7.

The load and displacement values are taken from the above figures to compute the fracture toughness values by using equation (4)

$$K_c = \sigma \sqrt{\pi a}$$  \(4\)

Where \(K_c\) is fracture toughness in KPa \(m\), \(\sigma\) is stress in KPa, and \(a\) is displacement in m.

The values of fracture toughness obtained for V-notch and plain specimens are given in table 1.

The test method involves three-point loading for simply supported notched beam for evaluating tensile strength of AlSiC using load-deformation curve. Using this test, the limit of proportionality (LOP) is determined, which is a point on the load-deformation curve. Two equivalent flexural strengths identify the material behavior up to this deflection. The flexural tensile strength is the highest stress experienced by the material at the time of yield. 5 specimens of 1 vol% SiC/Al were subjected to bending test and average is found to be 38.355 N/sq.mm, as shown in figure 8.

Values of tensile, yield, breaking, and flexural strengths are shown by error bars as shown in figure 9. Similarly, fracture toughness of V-notch and plain specimen is also represented as error bar in figure 10.

3.3. Fractography
Fracture surface of SiC reinforced Al composites after tensile loading is shown in figure 11(a). A significant number of dimples and cleavage facets can be observed resulting in the mixture of ductile and brittle fracture
during tensile loading. Ductile fracture occurs in Al matrix due to formation of voids causing growth of cracks. High stress concentration, decohesion, and work hardening of SiC particles are responsible for brittle fracture. SiC reinforced Al composites failed due to combination of these ductile and brittle fracture. Figure 11(b) shows the fracture surface of SiC/Al composites after fracture toughness test confirming the absence of voids or debonding between Al and SiC indicating better interfacial bonding. Microcracks can be observed in the fractured surface at the interface of Al matrix and SiC and progressed during impact loading. Fine dimples are also visible resulting in the ductile fracture of composites. Fracture surface after three-point bending test is shown in figure 11(c). Strong bonding between the interface of SiC and Al matrix is responsible for absence of voids and decohesion. Fracture mechanism is ductile due to the presence of dimples observed in the fractured surface.

3.4. Study of machining characteristics

The MMC composite is machined on a computer numerically controlled (CNC) milling machine for studying its machining characteristics as shown in figure 12. CNC milling machine removes the material using G-code and M-code to create various surface features. CNC machine can control some functions automatically such as motion of tool and workpiece, process parameters (feed, cutting speed, depth of cut etc.), turning coolant on/off etc. The input parameters taken for machining of new material obtained in alloy form is given in the table 2:

The regression models obtained from MINITAB software is as follows:

\[
\text{Flatness} = 25.56 - 0.001500 V - 0.03000 f + 1.67 d + 0.0667 Sr
\]

\[
\text{MRR} = -13.96 + 0.000271 V + 0.07048 f + 30.40 d - 0.1844 Sr
\]

\[
\text{TWR} = -0.1664 + 0.000002 V + 0.000562 f + 0.2709 d - 0.001320 Sr
\]

Linear regression models are used to predict the selected output variables (flatness, material removal rate (MRR), and tool wear rate (TWR)) from data of experimental results shown in table 3. An increase in Sr, v, and d decrease flatness, which is desirable. But flatness increases with feed rate, which is undesirable. Similarly, higher cutting speed increases both MRR and TWR. Hence for Al-SiC composites, machining at higher cutting speeds is required for better results.

The composite is also machined in an electric discharge machine, and the results suggested that surface finish is better for nano SiC reinforced aluminum composites when compared to micro SiC reinforced aluminum composites.
Figure 11. Fracture surfaces of SiC reinforced composites after (a) tensile test, (b) fracture toughness test, and (c) three-point bending test.

Figure 12. Final picture view of Al-SiC composite.
4. Conclusions

In this study, SiC is extracted from rice husk, and a composite material of Al with SiC (in nano form) is prepared using the stir casting method. Mechanical and Machining characteristics are studied for casted new material. The following conclusions are made:

1. Surface characteristics (XRD) reveal the presence of SiC in the MMC prepared, and SEM images show the agglomeration of large SiC particles in some spots.

2. Mechanical characteristics, such as tensile strength and hardness of the casted composite, are improved, and the material can be used for a variety of applications.

3. The fracture toughness at low temperature is very high, and the material is suitable for aerospace applications.

4. The machining characteristics reveal that the selected output characteristics (flatness, MRR, and TWR) for MMC made with nano SiC particles are better when compared to MMCs made with micro-sized SiC particles.

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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

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ORCID iDs

Madhusudan Baghel  https://orcid.org/0000-0001-7206-0828

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