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ORIGINAL PAPER

Mechanical Behavior and Metallographic Characterization of Microwave Sintered Al/SiC Composite Materials – an Experimental Approach

Shoba Chintada[1] · Siva Prasad Dora[1] · Dorathi Kare[2]

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Abstract

It is traditionally thought to be challenging to incorporate tougher ceramic particles into a softer aluminium matrix. Powder metallurgy has emerged as a significant fabrication technology in this context. Silicon carbide (SiC) is reinforced to aluminium with varying reinforcement, i.e., 5, 10, and 15 %. The samples were sintered in a microwave sintering furnace at 550 °C ± 5 °C. The scanning electron microscope and the field emission scanning electron microscope (FESEM) were used to inspect the microstructure (structure/shape, dislocations, and grain distribution) of prepared powders and sintered composites. Water displacement methods were used to assess the density and porosity of prepared composites. X-ray diffraction (XRD) is used to assess the existence of intermetallic compounds (if any) and contaminations. Mechanical investigations were performed on aluminium and its composites, as well as the effect of particle size on the mechanical properties of Al/15 %SiC were studied and presented. Density and porosity found to incline with the increase in SiC content. Properties such as hardness, ultimate tensile strength (UTS) and yield strength (YS) of the reinforced composites (5, 10, and 15 %) were significantly improved, whereas elongation rapidly declined compared to pure aluminium. Also, the UTS and YS increases with the decrease in reinforcement particle size. The dominant mechanisms are ascertained to be dislocation density, refined grain size, and porosity.

Keywords Aluminium · Mechanical behavior · Microwave assisted sintering · Powder metallurgy

1 Introduction

The mechanical properties of the reinforced composites were influenced by the random dimensions, shape, and weight fractions of the reinforcement, matrix material, and reaction at the junction of the interface. Reinforced material should be uniformly dispersed to focus on increasing mechanical properties such as micro hardness and tensile strength. According to the findings, diffusion bonding, particle size, and particle shape all have an effect on the microstructural integrity and mechanical performance of the desired compact [1]. Ceramic reinforced composites with apparent low density, higher melting points, elevated hardness, good Young's modulus, and resistance against corrosion are becoming more popular in advanced manufacturing, avionics, and ceramic armour materials [2].

Metal matrix composites (MMCs) are superior advanced materials that are created by combining two or more materials to achieve specific properties. They have gained popularity in all these years due to their superior strength quality, higher stiffness, and wear content when compared to unreinforced alloys [3–5]. Aluminium metal matrix composites (AMMCs) have been identified as potential candidates as typical MMCs due to their exceptional specific strength, wear resistance, and high-temperature performance. Massive research was done on ceramic based composites to analyze properties like resistance to wear, tensile structural behavior, fracture toughness, and cyclic stress behavior. Ceramic composites such as silicon carbide, rice husk ash, graphene, boron carbide, and carbon nanotubes outperform monolithic ceramics in terms of mechanical properties [6, 7]. Because of its unparalleled electrical, thermal, and mechanical properties, SiC has been widely used

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✉ Shoba Chintada
shobachintada@gmail.com

[1] Department of Mechanical Engineering, GITAM (Deemed to be University), 530045 Visakhapatnam, India

[2] Department of Mechanical Engineering, Sri Vasavi Engineering College, Tadepalligudem, A.P, India

as reinforcement in recent years to improve matrix performance [2, 8]. SiC particulates reinforced aluminium matrix composites (AMCs) require far more properties than conventional alloys, such as high strength, thermal properties, and frictional wear and corrosion resistance. Proper densification and microstructure which is free from micro-holes and other deficiencies is obtained when Al/SiC composites are sintered by SPS at 510 ºC [9, 10].

Powder metallurgy (P/M) provides homogeneous particulate distribution and causes less reaction between matrix and reinforcement in the production of AMMCs [11]. In recent years, powder metallurgical components have grown exponentially for use in the vehicle industry and related technologies. P/M has been found to be appealing because it achieves homogeneous particle distribution while processing AMMCs, and the method controls interface kinetics and works indefinitely at low temperatures. A high yield strength in the magnitude of 355 MPa, as well as UTS of 430 MPa, is achieved when SiC nano particles are dispersed uniformly into aluminium base matrix and sintered by hot iso-static press (HIP) sintering technique [12]. Microwave sintering, among the various sintering methods, provides extremely high heating rate, a shorter fabrication time, a homogeneously distributed morphology, and quality [13]. Microwave sintering of composites is found to be more efficient than traditional heating methods. Microwave heating, however, first produces heat inside the material and then heats the whole volume. Due to high heating rates and slow sintering temperatures, product uniformity, high yielding, homogeneous volumetric heating, minimal soaking time, environmental protection, equiaxed pores, effectual densification, refined microstructure, enhanced physical and mechanical characteristics, simplicity, distinctive properties, and reduced environmental hazards have been achieved [14–16].

From the economic analysis made by Venkatesh et al. [17] it is understood that microwave assisted sintering (MAS) is capable to produce high precision parts with reduced intermetallic compounds, with lesser defects, minimal sintering time and temperature. Several studies have revealed that sintered composites have also improved microstructural homogeneity and mechanical properties [18, 19].

A revolutionary integrated manufacturing approach is microwave processing of powder metallurgy metal composites has received little research attention in recent years. From a review made by Shoba et al. [20], it is recognized that, while developing sintered Al based composites various sintering parameters like pressure, temperature, time, sintering atmosphere (argon or nitrogen) play a crucial role. Although there is a lot of research on the microstructural behavior and mechanical characterization of Al/SiC microwave sintered MMC’s with varying SiC volume fractions, the effect of particle size on the mechanical behavior is rarely seen. Hence the present work addresses the microstructural and mechanical properties of pure Al and its composites, as well as the impact of three distinct particle sizes.

2 Experimentation

In this work, aluminum powder of 99 % purity and silicon carbide with 99.5 % purity are procured from Otto Chemie Pvt. Ltd., and are used as the starting materials. The powders are mixed in the proportion of 5, 10, and 15 % by weight in a ball mill (without balls). Next, the powders are compacted by applying 120 bar pressure using hydraulic press of 20 ton capacity [21]. Finally, the green compacted specimens were sintered at 550 °C for 90 min in a microwave heating furnace. The whole sintering process was performed under N_2 gas atmosphere [22, 23].

The experimental setup and the corresponding samples were presented in Fig. 1. With JEOL (JSM7100F) field emission scanning electron microscope (FESEM) the microstructural analysis were done on the samples which were cut in transverse direction. The samples for phase identification for the composites were characterized by X-Ray diffractometer (PROTO iXRD). Based on the Archimedes’ principle [24] experimental density of the samples is presented. The porosity of a composite can be obtained from the experimental and theoretical densities. Hardness of the microwave sintered aluminum samples was measured using a SHIMADZU G20 microhardness tester. A load of 500 gm was applied for 15 s, and the indentation diameter was measured at five different locations to measure the Vickers hardness number of the samples. Tensile tests of the sintered samples were performed on flat tension test samples collected with a width of 15 mm and a gauge length of 30 mm using a servo-hydraulic testing machine with a crosshead speed of 0.254 mm/min. The initial strain rate was $1.69 \times 10^{-4} s^{-1}$. SEM was also used to examine the fracture surface morphology of the specimens that were cut for the tensile test. In order to evaluate the effect of SiC particle size on the mechanical properties of the composites, SiC powder (37 μm) was milled (tungsten carbide balls of ϕ 10 mm) in a planetary ball mill for 5 and 10 h with a rotating speed of 250 rpm and ball/powder mass ratio of 10:1 to attain particle sizes of 23 and 10 μm respectively [25, 26]. The Al reinforced composites with different particle sizes are fabricated using the same conditions by powder metallurgical process to evaluate the tensile behavior.

3 Results and Discussions

The micrograph of the starting materials (silicon carbide and aluminium powder) considered for the present study is shown in Fig. 2. SiC particles with an average size of 37 μm were identified to be irregular and angular in shape. Aluminium

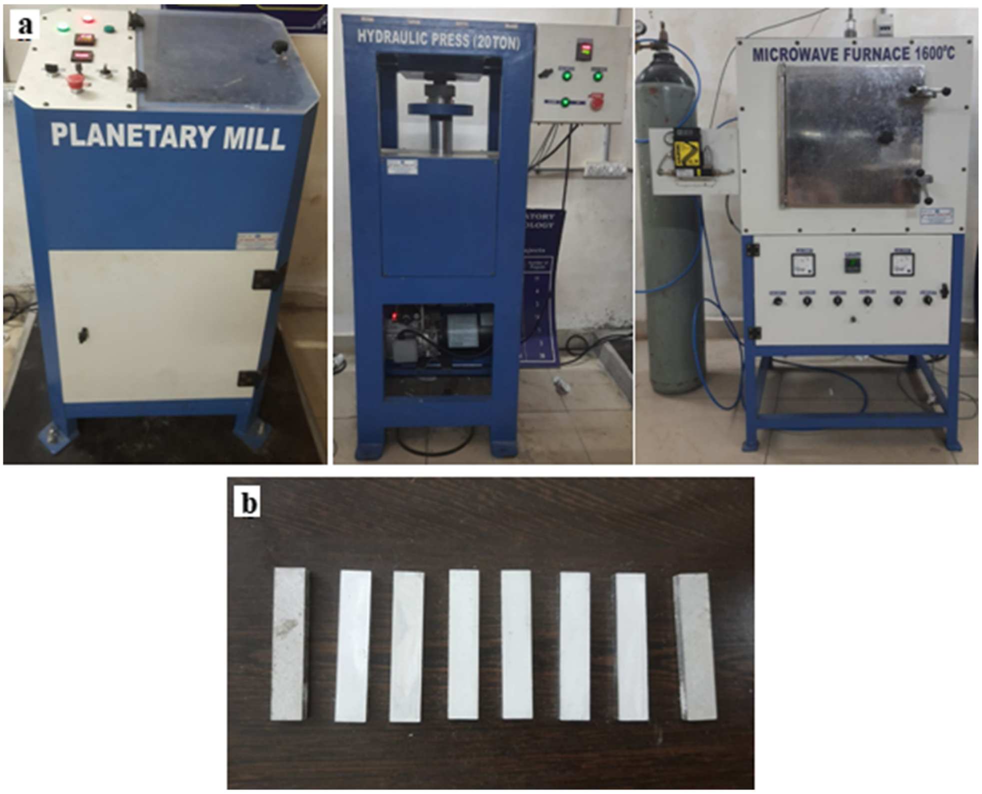


Fig. 1 (**a**) The experimental setup (**b**) fabricated samples

powder particles with an average size of 26 μm with elliptical-shaped particles were observed.

Figure 3 illustrates a scanning electron micrograph of microwave sintered samples of pure Al, Al + 5 %SiC, Al + 10 %SiC, and Al + 15 %SiC composites. A favorable distribution of strengthener particles in the matrix has been achieved indicating that the powder metallurgy with microwave sintering treatment effectively disperses the SiC particles throughout the aluminium matrix. The distribution of Al and SiC particles also seems to be heterogeneous. From Fig. 3b-d it can be seen that, the interfacial bonding between reinforcement and the matrix is extremely good. Figure 3e presents the EDX analysis, which confirms the presence of aluminium and silicon carbide in the prepared composites.

Figure 4 reveals the XRD pattern of the prepared composites. The presence of aluminium and silicon carbide peaks are acknowledged from the pattern. The XRD patterns of Al/SiC composites show that the diffraction peaks corresponding to SiC are steadily rising, indicating that the SiC particle content improves with the reinforcement ratio. It has been realized that SiC particles do not interact with aluminium to develop any intermetallic compounds such as the Al_3C_4 phase etc.

3.1 Density and porosity

Density measurements were made to determine the porosity of the prepared composites and the impact of the reinforced ceramic content on the composites' density. The density and porosity of the manufactured composites with diverse SiC particle contents are determined [24, 27]. Figure 5 depicts the variation in the composite material's measured density and porosity. Compared to pure aluminium, the density was

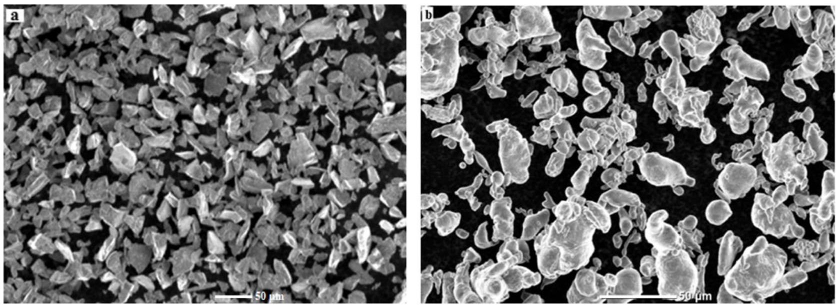


Fig. 2 a) SEM micrographs of (**a**) SiC particles (**b**) Al powder

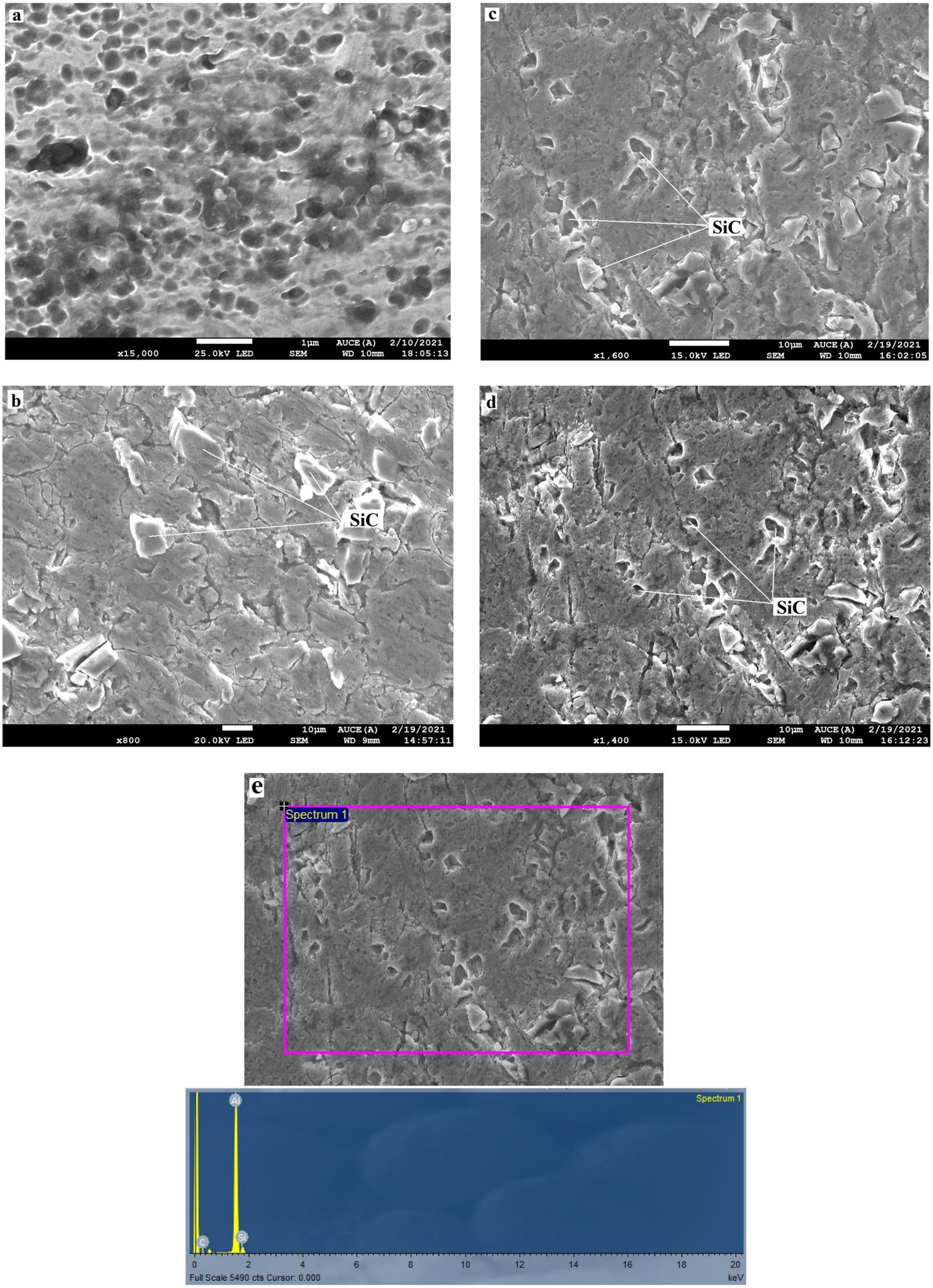


Fig. 3 The sintering data (power and piston movement) for (**a**) SiC-Al2O3, (**b**) SiC-TiC, and (c) SiC-B4C during the FSPS run

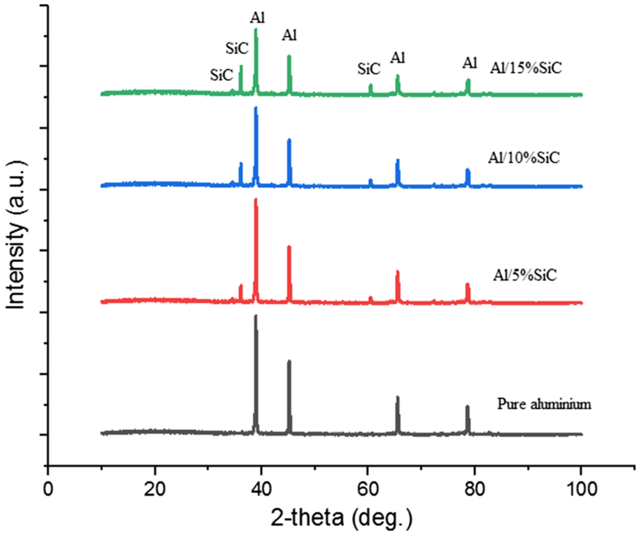


Fig. 4 XRD pattern of pure Al, Al/5 % SiC, Al/10 % SiC and Al/15 % SiC composites

increased by 6.28 %, 10.75 % and 16.66 % for composites containing 5 %, 10 %, and 15 % SiC particulates, respectively, which is due to the addition of higher denser SiC particles in aluminium. The porosity was determined [28] using known theoretical and experimental densities. The porosity of composites enhanced slightly with increasing SiC content. The maximum porosity obtained is 2.1760 % for Al/15 %SiC (Fig. 5). The amount of porosity in the produced composites is less than 3 %, which is really the utmost porosity level tolerable in the production of AMMCs [29]. This means that, despite having a higher porosity level, the porosity level of up to 2.1760 % is believed to be acceptable and appropriate in the current research work.

3.2 Hardness

Hardness refers to a material's resistance to surface indentation. Because of the inclusion of harder SiC particulates in the aluminium matrix, the hardness increases as the fraction of reinforcement increases. Figure 6 depicts the hardness of pure aluminium and its composites. The Vickers hardness value of pure Al is 93.5 VHN, and the hardness value rises as SiC content rises, with the maximum obtained hardness value seeming to be 121.5 VHN for Al/15 %SiC sintered composite. The existence of harder and more tightly bonded SiC particles in the Al matrix, which impedes dislocation movement, increases the hardness of AMMC's [30]. An increasing trend in the hardness of the composites signifies that the particulates present in the matrix has progressed the composite's overall hardness. As the matrix is a soft material and the reinforcement particulates are harder and make a positive contribution to the hardness of the composites. Also, the hardness of the reinforced sintered composites has improved due to a reduction in the grain size of the composites. It is also reported by the Hall-Petch relation that hardness inclines with a decline in grain size [31].

3.3 Tensile behavior

As per the tensile test results, the reinforced composites have a higher tensile strength than pure Al. The ultimate tensile strength (UTS) of composites was reported to increase with an increment in SiC content, as per the engineering stress-strain plots (Fig. 7). When compared to pure Al, the UTS for all the samples (5 %, 10 %, 15 %) were drastically improved

Fig. 5 Variation in density and porosity at varying reinforcement

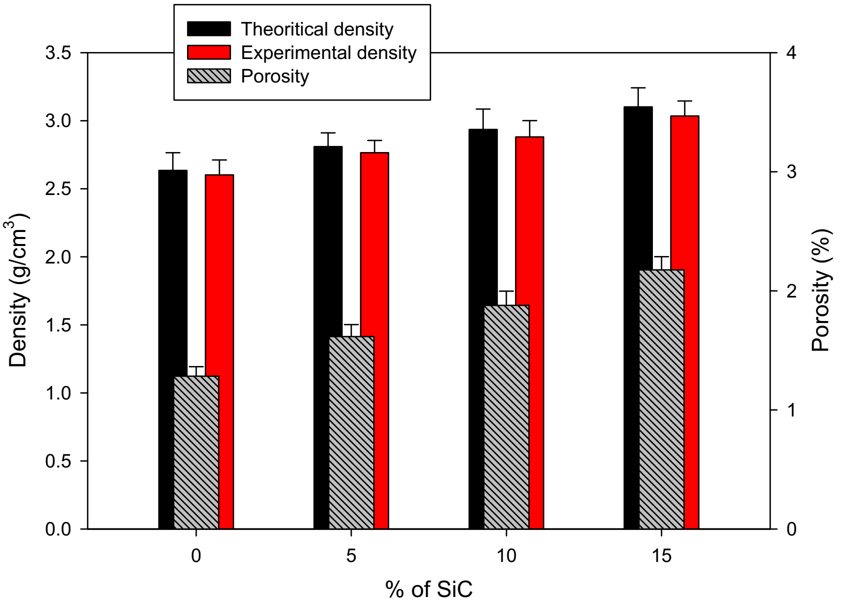
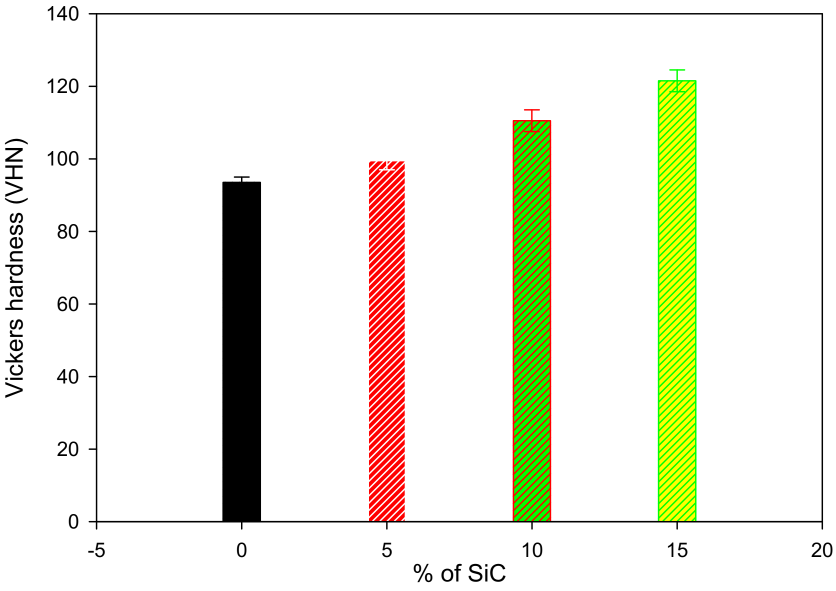


Fig. 6 Variation of hardness with varying SiC content in the composites



by 29.64 %, 71.59 % and 103.26 %, and that of yield strengths (YS) were 21.16 %, 49.36 % and 61.06 %, respectively, as depicted in Fig. 8.

Based on the studies carried out, many researchers proposed various strengthening mechanisms while performing investigations on AMMCs. Nonetheless, the mechanical strength of the composites is not dependent on a single mechanism but rather on a combination of several mechanisms that may act concurrently. In general, the strengthening mechanisms of MMC's include the Hall- Petch strengthening, the Orowan strengthening, thermal mismatch strengthening and load transfer from matrix to reinforcements [31, 32]. The identified strengthening mechanism in the present study is reported in the subsequent paragraphs:

3.3.1 Dislocations based strengthening mechanism

SiC has a coefficient of thermal expansion (CTE) of about 5 × 10^{-6}/°C, whereas aluminium has a CTE of about 23 × 10^{-6}/°C. Dislocations form at the interfaces due to the difference in CTE's [33]. In general, a mismatch in the CTE between the reinforcement and the matrix alloy results few dislocations at the junction of the interface, which strengthens the matrix alloy. The contribution of ceramic particles (SiC in this study) to total composite tensile strength could be attributed to higher

Fig. 7 Stress –strain plot for Pure Al, Al/5 %SiC composite, Al/10 %SiC composite and Al/15 %SiC composite

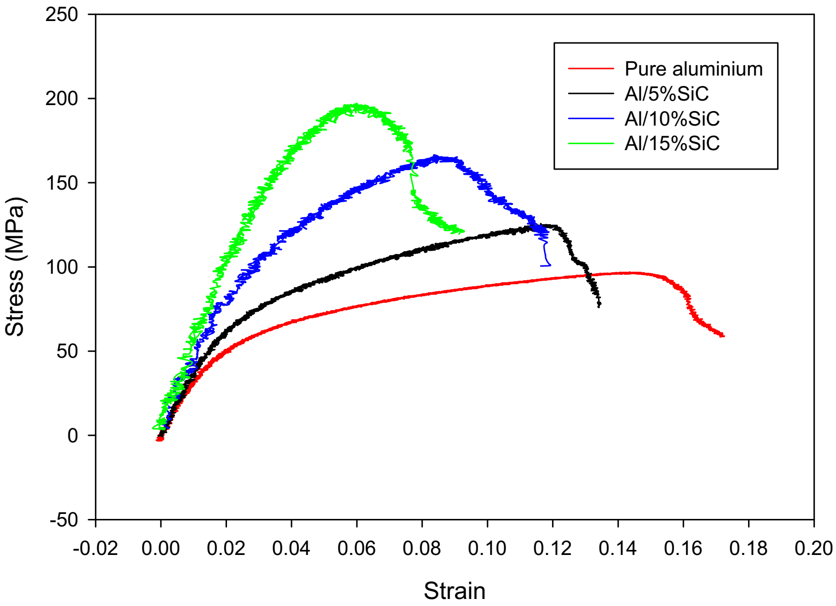
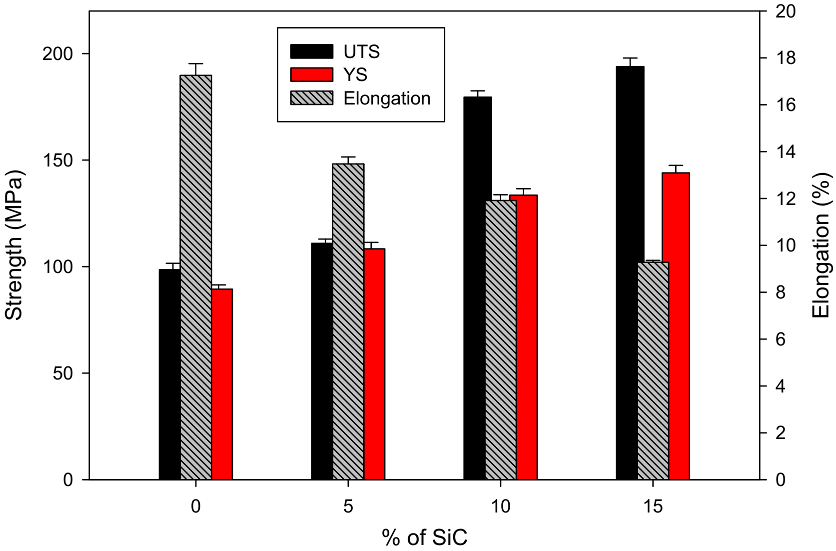


Fig. 8 Variation in ultimate tensile and yield strength of composite with varying SiC content in the composites



dislocation density in the composite matrix caused by a thermal mismatch during fabrication followed by sintering between the ceramic particulates and the Al matrix. Several studies [34–37] have found the same pattern of this behavior. Thermal strain is found to be related to the CTE of SiC and aluminium. The higher the dislocation density, the higher is the alloy's strength [38]. The relation between the strength and dislocations/dislocation density (ρ) due to the thermal mismatch during fabrication is stated by [39]:

$$\text{Delta}\sigma_y = Gb\sqrt{\rho} \tag{1}$$

Where, G is shear modulus.

It can be seen from Eq. 1 that the greater the number of dislocations, the greater the composite's strength. The number of dislocations will rise as the SiC content increases. It is obvious that the dislocations arises from the interface and appear to be large in number for Al/15 %SiC composites, resulting in higher strengths.

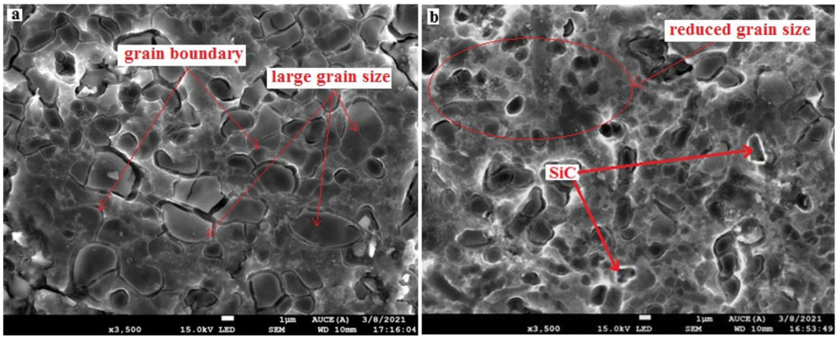
3.3.2 Strengthening Due to Refined Grain Size

Figure 9 presents FESEM micrographs of the surface of pure Al and Al/15 % SiC composite. This micrograph clearly shows that both the Al and the composites have a distinguishable grain boundary, but the grain size is smaller and there are more grain boundaries in Al/15 %SiC composites. Tensile strength is inversely related to average grain size [40]. The area resists tensile or external force as the grains are refined, and the number of boundaries multiplies. Because of the frequent changes in the direction of dislocations at grain boundaries, dislocation motion is slowed. Higher grain refinement tends to improve the grain boundaries for SiC particles and matrix material, potentially delaying SiC particle detachment from the matrix material while still applying tensile load. The Hall–Petch relationship is well-known to achieve high strength by reducing grain size. The Hall–Petch equation mathematically describes the relationship between yield stress and grain size as mentioned in Eq. 2 [31]:

$$\sigma_Y = \sigma_o + \frac{K_y}{\sqrt{d}} \tag{2}$$

Where σ_Y is the yield strength and d is the grain diameter.

Fig. 9 FESEM micrographs of (**a**) Pure Al (**b**) Al/15 %SiC composite



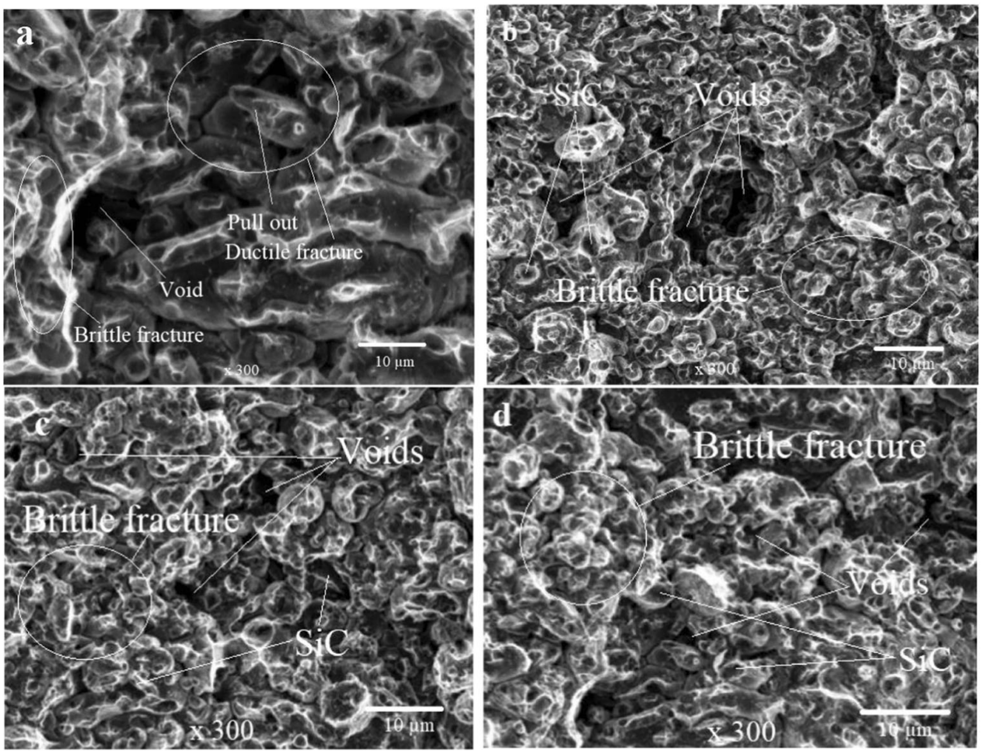


Fig. 10 FESEM images of fractured surface (**a**) Pure aluminium (**b**) Al/5 %SiC composite (**c**) Al/10 %SiC composite (**d**) Al/15 %SiC composite

From Eq. 2 it is realized that, the yield strength is inversely related to grain size. As can be seen in Fig. 9, as the SiC particle content increased, the grain size shrank dramatically, which results in higher strength for Al/15 %SiC composite.

3.4 Fracture Analysis & Elongation

The fracture morphology of tensile samples was analyzed to understand the effect of reinforcement particles in the fabricated composites. Corresponding FESEM images of fractured surface of the pure Al and the composites is shown in the Fig. 10. The fracture morphology of pure Al revealed typical ductile fracture characteristics for pure Al. Pull out of aluminium is clearly observed in fractured samples of pure aluminium, results in a cup and cone fracture, indicating ductile nature of the metal. Also, it is further noticed that the fractured surface consists of voids of considerable size. This feature is presented in Fig. 10a. The size of voids declined with increase in SiC particulate content in the composites. The grain refinement due to microwave sintering and the addition of SiC particulate content are the primary causes for the shrinkage of voids in the composites, as observed from the Fig. 10b-d.

Despite the fact that the size of the voids in the composites decreases significantly as the SiC particulate content of the composites increases, the presence of hard reinforcement particles in the soft matrix causes plastic flow localization at the Al/SiC interface. Hence, the fracture morphology of Al reinforced with 5 %, 10 % and 15 % SiC composites revealed brittle fracture characteristics. Also, the stress concentration at the matrix particles interfaces produces reduced elongation, which includes particle fracture at higher reinforcement percentages. The ductility is significantly impacted by the fabrication technique and perhaps even the reinforcement. Obviously, powder metallurgy followed by microwave sintering enhanced the UTS, but high percentage of SiC reinforcement (of 15 %) exhibited reduced ductility of 9.28 % when compared to pure Al which has an elongation of 17.25 %. The inclusion of reinforcement content reduces the elongation fracture of the composites [41–43]. Similar studies [42–48] were considered for comparison of the present work and the same is plotted for a quick view in Fig. 11. The fracture of reinforcement particulates and localization of matrix deformation are thought to be the primary causes of composite ductility reduction. Furthermore, the elongation was greatly reduced as the SiC content in the composites increased in comparison to pure Al, resulting in an increase in hardness and tensile strength. Similar results were presented for various fly ash reinforcement contents [48].

3.5 Particle's Size Effect on the Mechanical Properties of Al/15 %SiC Composites

It is obvious from the experiments that 15 % SiC reinforced composite demonstrate increased strength; therefore the effect of SiC particle size on 15 % SiC reinforced composite has been explored. The tensile properties (UTS, YS and elongation) of Al/15 %SiC composite with 37 µm, 23 and 10 µm particle sizes of SiC are illustrated in Fig. 12. It is clear to

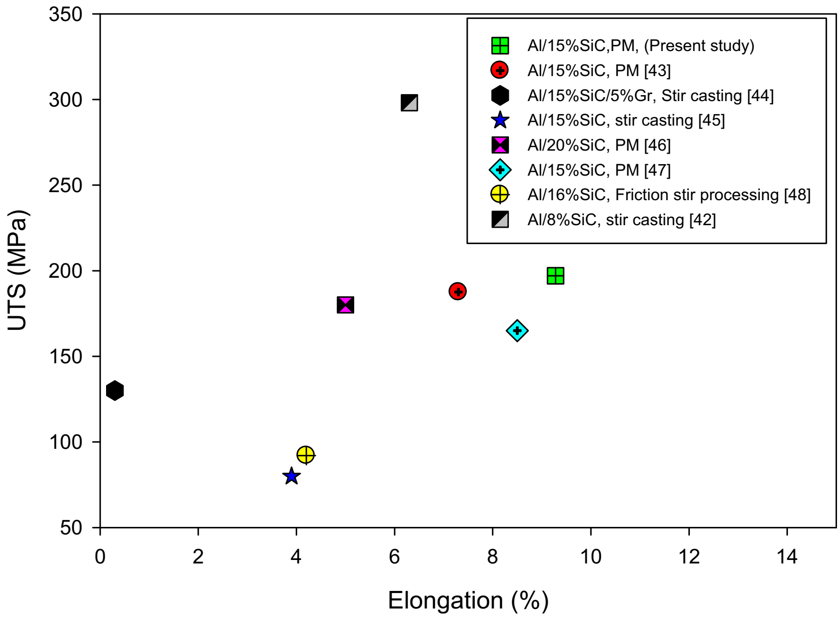


Fig. 11 Comparison of ultimate tensile strength (UTS) and elongation between the present work and other similar works [42–48]

observe that UTS, YS and elongation of Al/15 %SiC composite increase with the decrease in the particle size, indicating that particle size has substantial impact on the composite's mechanical behavior. Owing to the smaller inter-particle spacing and higher work hardening rate, it is widely acknowledged that minimizing the reinforcement size can result in a finer microstructure and enhanced mechanical characteristics of composites for a specified particle volume fraction [41]. The effects of direct and indirect strengthening are both enhanced when particle size is reduced. It can be seen from the stress-strain curve for varying particle sizes of SiC (Fig. 13) that the elongation of the composites increases with the reduction in particle size. This could be because, the inter-particle spacing decreases when the SiC particulate size lowers for a fixed particle volume percentage. The interface area between the matrix and the SiC particulates grows as the SiC particle size decreases, allowing more load to be transmitted from the matrix to the SiC particulates. It's worth noting that a big interfacial area might help the matrix generate more dislocations, which improves the mechanical properties of the composites.

Larger particulates are more easily fractured than smaller ones. This is very much visible from the corresponding FESEM images (Fig. 14) of fractured surface of Al/15 %SiC composites with 37 µm, 23 and 10 µm particle sizes of SiC. Figure 14a-c represents voids that range from large to small size. It is understood that the particle fracture strength is controlled by the intrinsic voids within the particulate. Because the size of a void is restricted by the particle's size, larger particles are more prone to fracture. Hence, the elongation is greatly improved for Al/15 %SiC composites with

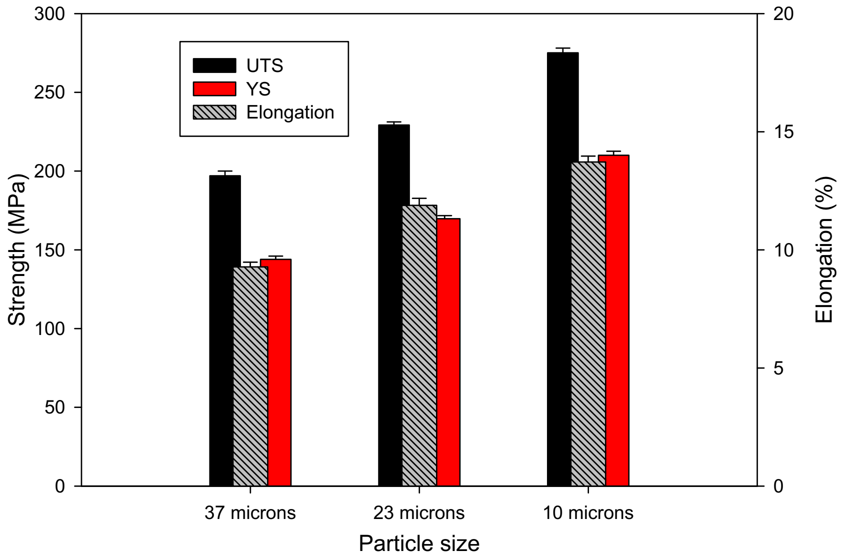


Fig. 12 Variation of UTS, YS and elongation of Al/15 %SiC composites with 37 µm, 23 and 10 µm particle sizes of SiC

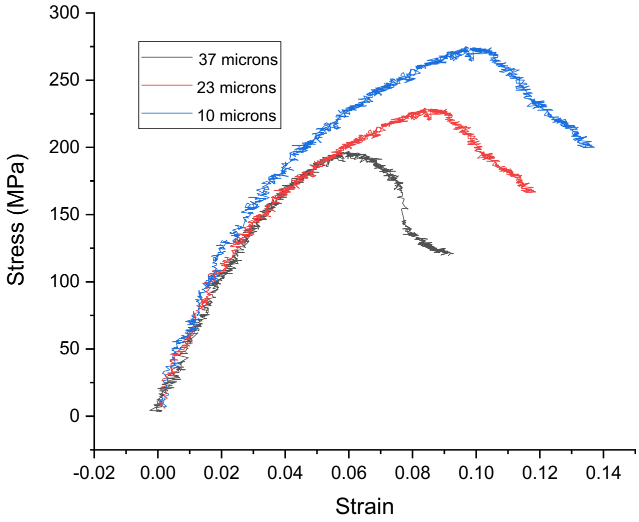


Fig. 13 Stress –strain plot of Al/15 %SiC composites with 37 μm, 23 and 10 μm particle sizes of SiC

10 μm (13.72 %) particle sizes of SiC when compared to 37 μm (9.28 %) and 23 μm (11.88 %).

4 Conclusions

The powder metallurgy technique with microwave sintering mode was realized to be effective in dispersing the SiC particles throughout the aluminium matrix. The density of Al/15 %SiC composites was raised by 16.66 %, when compared to pure aluminum. The Al/15 %SiC composite achieves a maximum porosity of 2.18 %, which is substantially within acceptable limits. The Al/15 %SiC composite exhibited an improved UTS, YS, microhardness while having a low elongation. Increased dislocation density and refined grain size are the possible mechanisms that are responsible for higher UTS and YS. The results reveal that particle size has a substantial impact on the composites mechanical properties. For Al/15 %SiC composite the UTS, YS and elongation were found to increase with the decrease in particle size. In comparison to the Al/15 %SiC composite with 10 μm sized SiC particulates, there was a 39.6 % and 45.8 % rise in UTS and YS, respectively compared to 37 μm sized SiC particulate composite.

Authors' Contributions Chintada Shoba: conceptualization, methodology, writing, D. Siva Prasad: Editing, Supervisor, Dorathi Kare: Experimentation and characterization.

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Data Availability As this manuscript is a part of ongoing research at this point of time data cannot be shared.

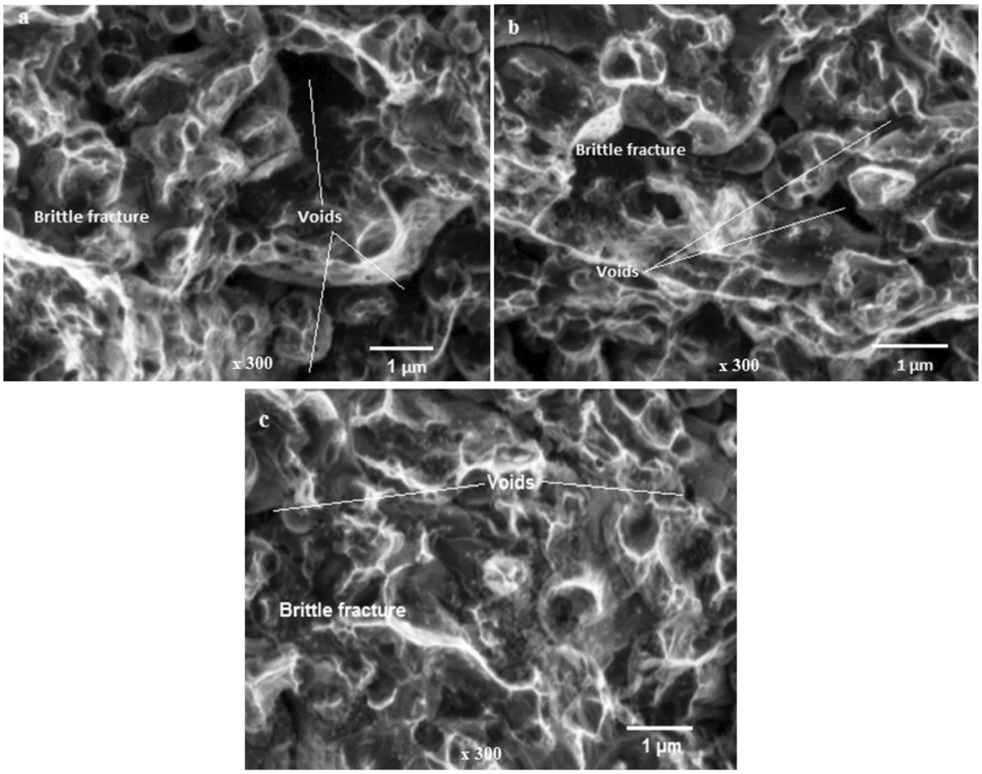

Fig. 14 Fracture SEM images of Al/15 %SiC composites at different particle sizes of SiC (**a**) 37 μm (**b**) 23 μm (**c**) 10 μm

Declarations

Conflict of Interest All authors declare that there is no conflict of interest.

Ethics Approval and Consent to Participate The manuscript does not report on or involve the use of any animal or human data or tissue and hence not applicable.

Consent for Publication Yes.

Competing of Interest All authors declare that they have no competing interests.

Consent to Participate Not applicable.

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