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ORIGINAL ARTICLE

Characterization of microstructural and mechanical properties of hybrid Al/SiC/Al_2O_3 nanocomposites

Penchal Reddy Matli[1] · **Guttikonda Manohar**[2] · **Rokaya Abdelatty**[3] · **Rana Abdul Shakoor**[3] · **Abdul Azeem**[4] · **Siva Sankara Reddy Lingala**[1] · **Rama Gopal Kotalo**[1] · **A. M. A. Mohamed**[5]

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Abstract

This study focuses on the fabrication of aluminum-based hybrid metal matrix composites with a consistent SiC content of 2 wt% and varying concentrations of Al_2O_3 nanoparticles (2, 4, and 6 wt%), aiming to understand their improved properties and explore their potential applications in diverse industries. By utilizing the powder metallurgy method, Al-SiC-Al_2O_3 nanocomposite samples were synthesized through microwave sintering, followed by a comprehensive examination of their microstructural and mechanical properties. Experimental results revealed a uniform dispersion of Al_2O_3 nanoparticles in the Al matrix. The microwave-sintered Al-2SiC-4Al_2O_3 hybrid nanocomposite demonstrated significant improvements in various mechanical properties compared to pure Al, including an impressive increase in microhardness by approximately 115% (86 HV vs. 40 MPa), a substantial enhancement in yield strength by approximately 54% (113 ± 4 MPa vs. 73 MPa), and a remarkable increase in ultimate compressive strength by approximately 19% (364 ± 3 MPa vs. 305 MPa). The uniform dispersion of dense alumina nanoparticles, collectively contributing to a combined effect of strengthening mechanisms, attributed the observed enhancement in the mechanical properties of the nanocomposites.

Keywords Aluminum · Al_2O_3 · Hybrid nanocomposites · Microwave-assisted sintering · Microstructure · Compression behavior

1 Introduction

Significant advancements have been made in materials science, particularly in developing novel lightweight composite materials that exhibit exceptional performance characteristics. In recent years, researchers have made extensive efforts to develop new materials that meet the diverse requirements of manufacturing industries, including increased strength, reduced weight, and cost-effectiveness. Composite materials have emerged as the preferred choice due to their superior properties compared to base alloys, leading to their widespread utilization in various sectors such as marine, aerospace, defense, and automobile industries [1, 2]. Moreover, composites incorporating multiple reinforcement materials have shown great potential, enhancing hardness while reducing material costs [3].

Aluminum (Al)-based metal matrix MMCs (Al-MMCs) have become prominent in the areas of aerospace and automotive industries due to their unique properties, such as low density, lightweight, high strength to weight ratio, high thermal stability, and superior wear resistance [1–4].

The incorporation of micron-sized particles as reinforcements in composites has demonstrated remarkable improvements in their properties [5]. The rise in the concentrations of hard ceramic reinforcements in the matrix material also decreases the ductility nature of the composite. Researchers found that utilizing nanoscale particles resulted in superior enhancement in the mechanical behavior of the composites

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✉ Penchal Reddy Matli
drlpenchal@gmail.com

[1] Department of Physics, Sri Krishnadevaraya University, Anantapur 515003, India

[2] Department of Mechanical Engineering, National Institute of Technology, Warangal 506004, India

[3] Center for Advanced Materials (CAM), Qatar University, 2713 Doha, Qatar

[4] Department of Physics, National Institute of Technology, Warangal 506004, India

[5] Department of Metallurgical and Materials Engineering, Faculty of Petroleum and Mining Engineering, Suez University, Suez 43512, Egypt

due to their high surface-to-volume ratios [6]. In structural applications, lightweight metal matrix composites reinforced with nano-sized particles offer superior mechanical performance and energy efficiency because of their lower density and superior specific strength and stiffness [6, 7]. Compared to microparticles, nano range reinforcement particles exhibit better performance due to more particles that create more interface regions. The formed interfaces hinder the dislocation movements and activate the dislocation strengthening mechanisms near grain boundaries, which ultimately enhances the robustness of the composite material by sacrificing the ductility of the material. The selection of a suitable reinforcement with optimum particle size in a composite plays a crucial role in the material's overall performance [8].

Hybrid composite materials have the ability to work under higher pressure and temperature and have various applications [9–11]. Aluminum hybrid metal matrix composites (AHMMCs) are the new generation of metal matrix composites (MMCs) and have become very popular as they exhibit multifunctionality and appropriate properties such as superior strength, wear resistance, low weight as well as good stiffness, high hardness, and low manufacturing cost [12–14].

For the development of Al-based hybrid composites, various reinforcements like SiC, Al_2O_3, AlN, ZrO_2, Y_2O_3, SiO_2, TiO_2, Si_3N_4, B_4C, BN, TiC graphene, and CNT were used to mixed with Al [15–23]. The incorporation of SiC and Al_2O_3 nanoparticles into Al-MMCs has attracted considerable attention due to their potential to improve performance and offer economic advantages [24]. The content of the reinforcements primarily influences the weight percentage of these composites. In hybrid composites, the utilization of SiC and Al_2O_3 as reinforcements is prevalent due to their exceptional properties. Compared to monolithic ceramics and ceramic composites, hybrid ceramics demonstrate a notable enhancement in mechanical properties [25].

Various manufacturing methods have been adopted to fabricate Al-based nanocomposites, such as squeeze casting, powder metallurgy, in situ, stir casting, and SPS (spark plasma sintering) [9, 15, 17, 23]. Each synthesis process possesses different advantages. Among these, the powder metallurgy (PM) technique is selected for synthesizing AHMMCs, owing to its cost-effectiveness, improved properties, and environmentally friendly production method [19].

The powder metallurgy process comprises three fundamental steps: (i) powder blending, (ii) compaction, and (iii) sintering. A planetary ball mill is utilized throughout the blending process to ensure the homogeneous distribution of reinforcements within the matrix material. The compaction of billets is performed at room temperature. The novel microwave technology can utilize faster sintering for both monolithic and composite powder compacts compared to conventional methods, resulting in materials with improved microstructural and mechanical properties. Further, a rapid heating rate and higher sintering temperatures can enhance densification, better activate the bulk transport processes, and produce near-dense bulk nanocomposites compared to using either microwaves or resistance heating individually [26]. Also, microwave sintering helps achieve better end-application properties with a massive reduction in processing time (~ 80%) and costs, as well as eliminates the need for an inert protective atmosphere, unlike in conventional sintering. Hence, microwave sintering can be considered economically viable for industries and environmentally friendly for CO_2 emission reduction [15].

Several powder metallurgy studies have been conducted on aluminum composites with a single reinforcement to investigate their mechanical and physical properties [19–21]. Adjusting the sintering temperature, time, and heating rate can alter the composite material's density and mechanical behavior [16]. In addition, the size and volume of reinforcement particles significantly influence the physical and mechanical properties of Al-based composites.

Nayyak et al. [17] employed the powder metallurgy process to develop Al-based hybrid composites using SiC and Al_2O_3 as reinforcements. They concluded that the sintering condition of 700 °C for 60 min was suitable for the aluminum metal matrix composites to achieve a higher density and improved mechanical properties. Dwivedi et al. [18] developed a hybrid composite by incorporating SiC and Al_2O_3 particles through a conventional stir casting process. They predicted that enhanced mechanical behavior would be optimum for 7.5% SiC and 7.5% Al_2O_3 levels. Rajesh et al. [19] successfully synthesized Al7075-SiC/Al_2O_3 hybrid aluminum metal matrix composites with 10% weight of SiC and Al_2O_3 reinforcements, attaining superior mechanical and tribological properties. Ramya et al. [20] carried out tensile and compressive behavior of as-cast and heat-treated AA2024 alloy hybrid composites reinforced with SiC/Al_2O_3 through the compo-casting method. The fabricated composites exhibited enhanced compressive and impact strengths in their mechanical behavior, but the heat treatment process reduced both hardness and ultimate tensile strength (UTS).

This study aims to analyze the morphology, reinforcement ratio, interface chemistry, and robustness of the Al/SiC/Al_2O_3 hybrid nanocomposite fabricated using powder metallurgy techniques assisted with microwave sintering, filling a gap in existing research.

2 Experimental procedure

2.1 *Materials and synthesis of Al/SiC/Al_2O_3 hybrid nanocomposites*

The matrix material used in this study is pure Al, while SiC and Al_2O_3 are selected as reinforcements to fabricate Al/SiC/Al_2O_3 hybrid nanocomposites. Table 1 describes the matrix and reinforcement particles, including their purities, sizes, suppliers, and densities. Table 2 presents the compositions of the Al/SiC/Al_2O_3 hybrid nanocomposites in terms of weight percentages (wt%).

For the production of composites, powder metallurgy approach [15] involving ball milling and microwave sintering was used. We precisely weighed aluminum, silicon carbide, and alumina powders using an analytical balance measuring kit (Sartorius, ENTRIS64-1S). To uniformly disperse the reinforcement particles in the matrix material, ball milling was implemented at a speed of 200 rpm for 2 h. The 1.0 g of composite powder was transformed into pellets by applying a pressure of 50 MPa for 60 s. Prepared pellets were microwave sintered at 550 °C with a heating rate of 10 °C per min for 30 min.

2.2 *Characterization of Al/SiC/Al_2O_3 hybrid nanocomposites*

The densities of the fabricated composites were determined experimentally using the Archimedes method, which involves measuring the displacement of a fluid when the composite sample is submerged. Theoretical densities were also calculated using the rule of mixtures, which considers the densities of the individual components and their volume fractions in the composite. X-ray diffraction (XRD) analysis was conducted using a PANalytical X'pert Pro X-ray diffractometer, with a scan step size of 0.01313°, to identify and characterize the secondary phases present in the composite material. Elemental mapping and dispersion analysis of the reinforcement particles in the matrix material were performed using energy dispersive spectroscopy (EDS) analysis, carried out with a field emission scanning electron microscope (FESEM-FEI Nova NanoSEM 450) equipped with an EDS detector, to analyze the dispersion of reinforcement particles within the matrix material.

Table 1 A description of matrix and reinforcement particles

S. no	Particles	Purity (%)	Size	Supplier	Density (g/cc)
1	Al	99.5	7–15 µm	Alfa Aesar	2.70
2	SiC (β-phase)	--	44–55 nm	Alfa Aesar	3.21
3	Al_2O_3 (γ-phase)	99+	20 nm	Alfa Aesar	3.95

Table 2 Composition of Al/SiC/Al_2O_3 hybrid nanocomposites (wt%)

S. no	Al	SiC	Al_2O_3	Compositions
1	100	0	0	Pure Al
2	98	2	0	Al-2SiC
3	96	2	2	Al-2SiC-2Al_2O_3
4	94	2	4	Al-2SiC-4Al_2O_3
5	92	2	6	Al-2SiC-6Al_2O_3

The hardness of the Al/SiC/Al_2O_3 hybrid specimens was evaluated using a Vickers hardness tester with a 50 gf load applied for 10 s as per the ASTM standard E384-08, and the representative sample hardness was determined by averaging ten hardness readings. The compression behavior of the composite was studied at room temperature using a Universal Testing Machine (Lloyd-Ametek LR50K Plus, USA) in accordance with the ASTM E9–89a with a strain rate of $8.3 \times 10^{-4}\ s^{-1}$. Each test was performed three times for statistical significance, and the compression stress–strain curves were used to measure the 0.2% offset compression yield strength (CYS), failure strain, and ultimate compressive strength (UCS).

3 Results and discussion

3.1 *Structural behavior of Al/SiC/Al_2O_3 hybrid nanocomposites*

The difference in density and porosity of microwave-sintered pure Al and Al/SiC/Al_2O_3 nanocomposites is depicted in Fig. 1. Observably, the experimental density of hybrid composites is greater than that of the pure Al matrix, and it will increase with the inclusion of hybrid reinforcements due to

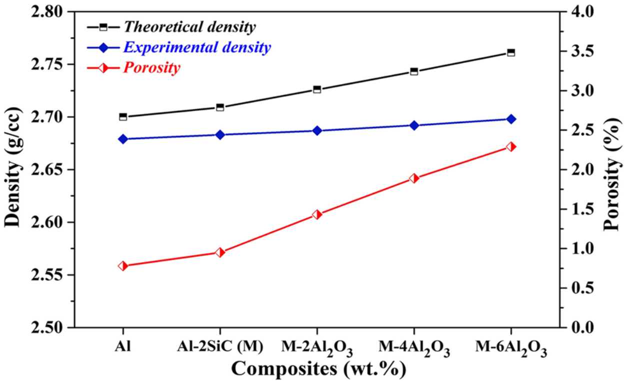

Fig. 1 Density and porosity of Al/SiC/Al_2O_3 hybrid nanocomposites

the presence of higher density reinforcements (SiC-3.21 g/cc and Al_2O_3-3.95 g/cc) in the base matrix (Al-2.7 g/cc).

Similarly, the increment in porosity is due to the formation of pore nucleation and an excess amount of reinforcements in the matrix [27, 28]. Similar observations were made by Mattli et al. [29]. The higher density and porosity are the properties that influence the mechanical and structural behavior of the materials.

Figure 2a depicts the X-ray diffractograms of added Al, Al_2O_3, and SiC powders, while Fig. 2b represents the microwave-sintered Al/SiC/Al_2O_3 hybrid nanocomposite peaks. The XRD analysis detected all the corresponding peaks of the materials, leading to the conclusion from the observations.

Due to the shorter microwave sintering time, XRD patterns were not detected in any impurity phases. The XRD peak planes of the Al matrix exist at (111), (200), (220), and (311), and the reinforcements SiC exist at (111) and Al_2O_3 exist at (400), which are well matched with JCPDS card no. 29-1129 (SiC) and 10-0425 (Al_2O_3). Due to their low weight percentage (%), the intensity of the reinforcement (SiC and Al_2O_3) peaks is negligible relative to the matrix and may be below the detection limit of the XRD technique [30].

Several investigations into microwave-sintered Al composites showed no impurity phases [31]. Due to the short sintering time, low sintering temperature, and rapid heating rates, the probability of secondary phase formation at interface regions is minimal during microwave sintering.

Field emission scanning electron microscopy was utilized to analyze the microstructural characterization of aluminum hybrid nanocomposites by altering the weight percentage of Al_2O_3 (2%, 4%, and 6%) while maintaining the weight percentage of SiC at 2%, which is shown in Fig. 3. The reinforcements (SiC and Al_2O_3) were found to be spread out evenly in the aluminum matrix. As shown in Fig. 3d, alumina containing 6 wt% of particles aggregated due to a larger concentration of alumina particles in the aluminum matrix [32]. The agglomeration formation affects the mechanical bonding between the matrix and reinforcements. The microstructure of the hybrid nanocomposite samples mainly consists of four regions: (i) grey color represents aluminum matrix, (ii) dark grey color represents SiC (black arrows), (iii) white particles represent Al_2O_3 (white arrows), and (iv) black patches are indicative of pore nucleation (red arrows).

Figure 4 shows the EDX spectrum of the hybrid Al/SiC/Al_2O_3 nanocomposites made by changing the weight percentage of Al_2O_3 while keeping the weight percentage of SiC the same. Figure 5a–c depicts the elemental mapping of Al/SiC/Al_2O_3 hybrid nanocomposites, which was conducted to better understand the elemental distribution. The presence of the main elements Al, Si, C, and O confirms the presence of Al_2O_3 and SiC nanoparticles and their uniform distribution in the Al matrix.

3.2 *Mechanical behavior of Al/SiC/Al_2O_3 hybrid nanocomposites*

Figure 6 represents the hardness values of the Al/SiC/Al_2O_3 hybrid nanocomposites. It is observed that with the addition of hard ceramic particles in the Al matrix material, the hardness values of the composite keep increasing with reinforcement levels with the aid of strengthening mechanisms. Maximum hardness values are obtained for 2% SiC + 4% Al_2O_3 combination; after that, hardness values decline due to agglomerates. An increase in the microhardness of developed hybrid nanocomposites (Fig. 6) is due to the presence of a homogeneous distribution of

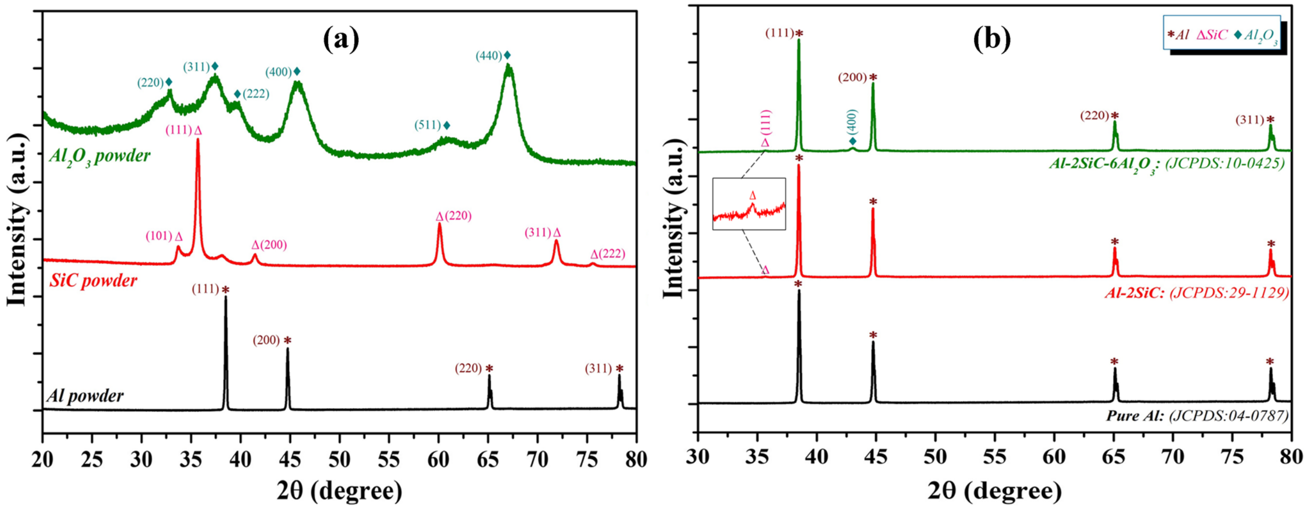

Fig. 2 XRD spectra pattern of **a** Al, SiC, and Al_2O_3 powders and **b** Al/SiC/Al_2O_3 hybrid nanocomposites

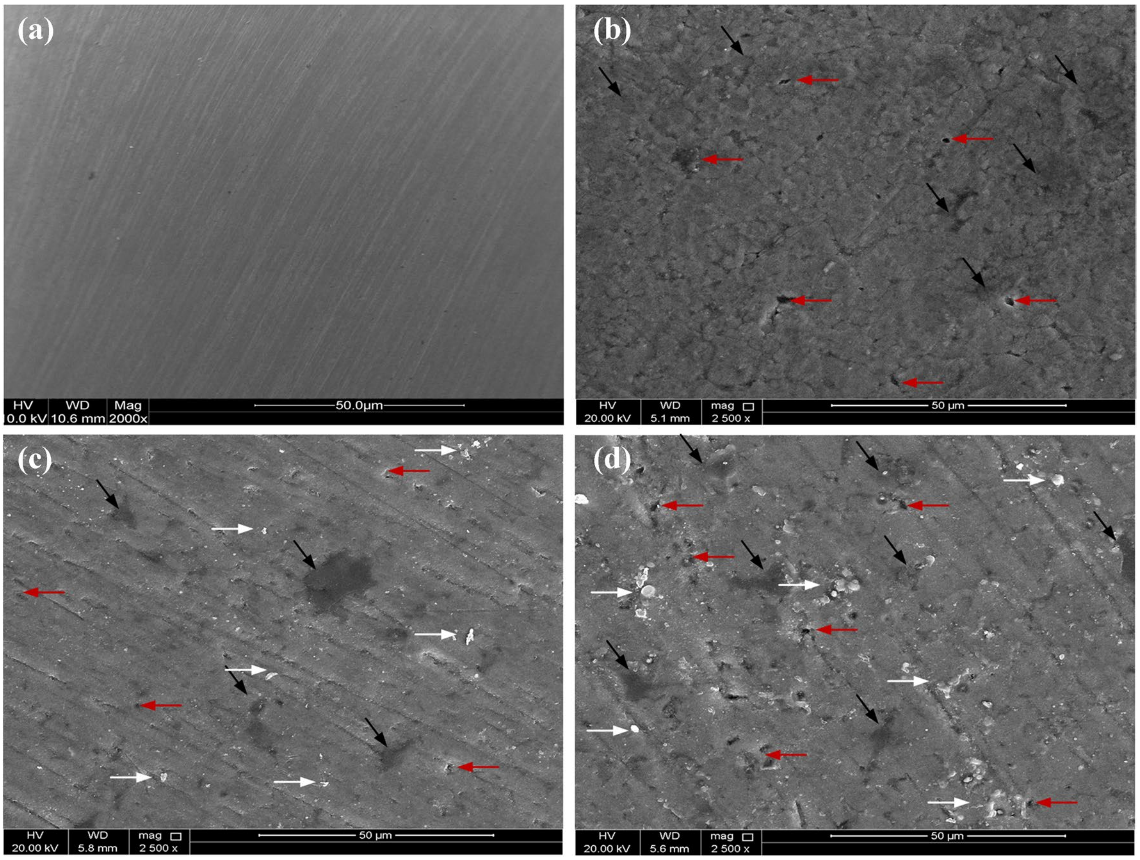

Fig. 3 FE-SEM images of the Al/SiC/Al_2O_3 hybrid nanocomposites. **a** Pure Al, **b** Al/2SiC, **c** Al/2SiC/4Al_2O_3, and **d** Al/2SiC/6Al_2O_3

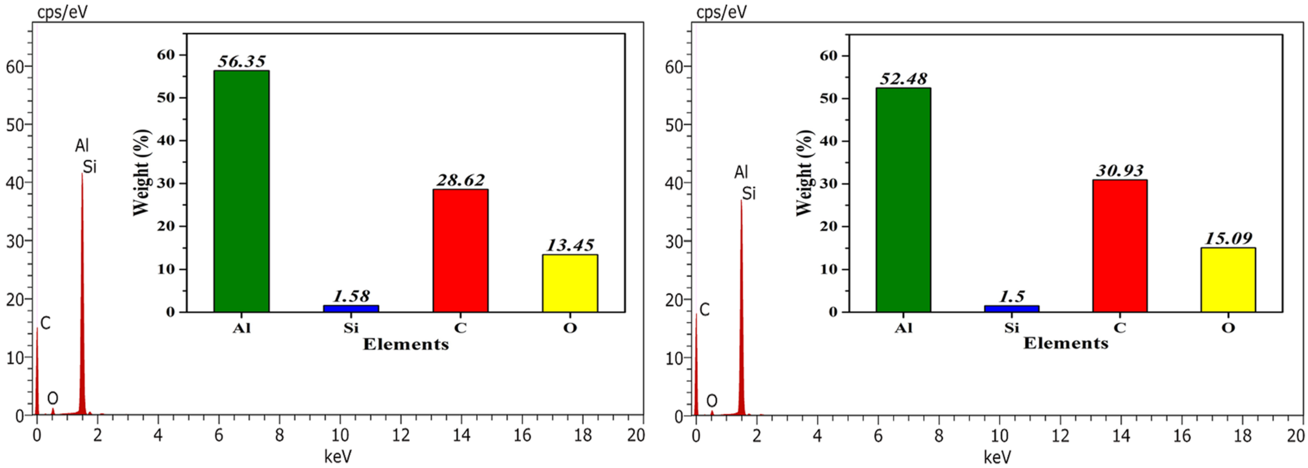

Fig. 4 EDX spectrum of **a** Al/2SiC/2Al_2O_3 and **b** Al/2SiC/6Al_2O_3 hybrid nanocomposites

reinforcement in the matrix. In addition, it is found that increasing the weight percentage of Al_2O_3 resists dislocation movement, resulting in a higher hardness when compared to monolithic aluminum alloy [33].

According to some reports, increasing the amount of nano reinforcement levels in the matrix material reduces the space between adjacent reinforcement particles, raising the stress levels required for dislocations to move between the

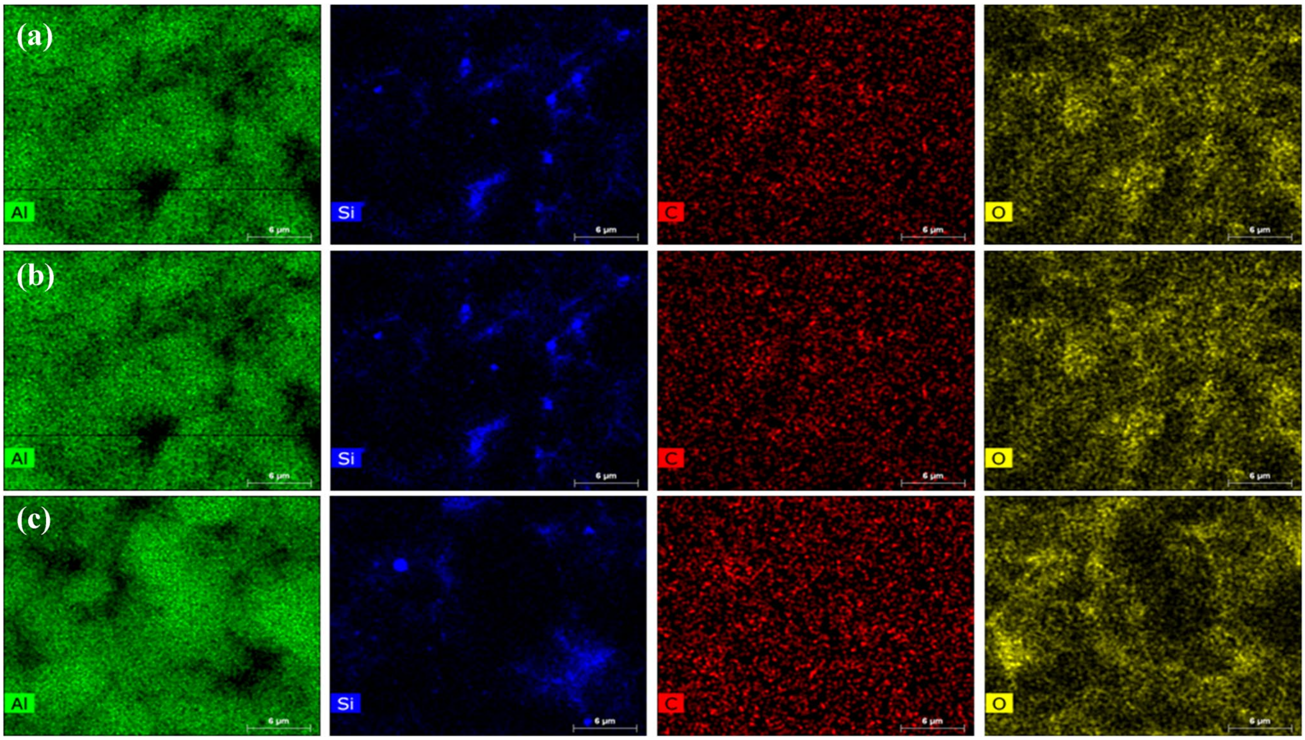

Fig. 5 Elemental mapping of **a** Al/2SiC/2Al_2O_3, **b** Al/2SiC/4Al_2O_3, and **c** Al/2SiC/6Al_2O_3 hybrid nanocomposites

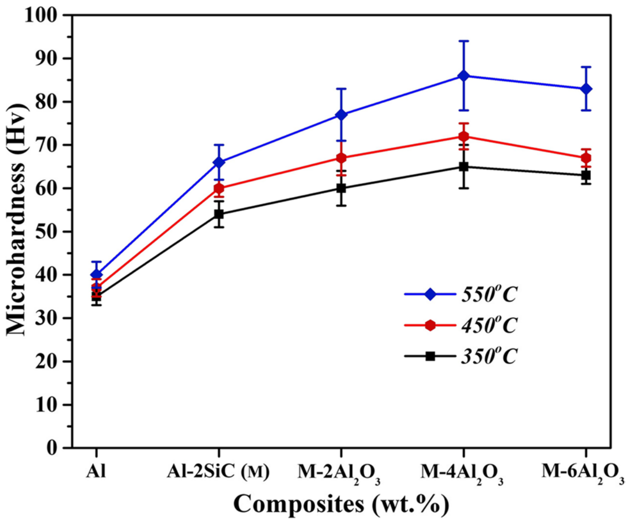

Fig. 6 Impact of sintering temperature on the hybrid Al/SiC/Al_2O_3 nanocomposites' microhardness

reinforcement particles and decreasing the ductility of the composite material. The Orowan strengthening mechanism plays a major role in strengthening the composite material.

Some investigations show particles smaller than ten microns should contribute to the Orowan strengthening mechanism [34, 35]. In this view, the present fabricated composites assist with Orowan strengthening mechanisms. The even distribution of reinforcement particles in the matrix material can also activate a dispersion strengthening mechanism that activates efficient load transfer mechanisms between the matrix and reinforcements through strong interface bonds created by the microwave sintering mechanism. The presence of nanoscale particles also helps restrict grain growth during the sintering process through the Zener pinning effect [36]. Additionally, it is noteworthy that the microhardness of Al/SiC/Al_2O_3 nanocomposites increased at 550 °C compared to 350 °C, attributed to the enhanced particle-matrix interfacial bonding at the higher sintering temperature [37]. Manohar et al. observed similar results while studying AA7075/SiC/graphite hybrid composites sintering at various microwave temperatures [38]. Additionally, adopting microwave energy restricts the rapid grain growth phenomenon with its unique features. This restriction is caused by the rapid diffusion process initiated at low temperatures due to the presence of high-density lattice defects generated during the ball milling process [39]. The unique feature of internal heat generation from the inner core of the particle reduces the thermal mismatch phenomenon at interface regions, allowing accumulation of dislocations at interface regions to cause optimized enhanced dislocation density mechanism [40]. Another advantage of microwave sintering is that as reinforcements act as heat generators, nanoreinforcement agglomerates in the matrix material act as hotspots and create strong interface bonds with adjacent matrix particles.

Examining the compressive behavior of Al/SiC/Al_2O_3 hybrid composites is crucial due to its specific relevance to applications, potential variations in material strength under diverse loading conditions, and the critical insights it provides into failure modes associated with compression. In some instances, the compressive behavior may even outweigh the importance of tensile behavior. While tensile properties are undoubtedly significant, a thorough analysis of compressive behavior ensures a comprehensive understanding of the material's mechanical performance, aiding in informed material selection and design decisions tailored to specific applications. Figure 7 shows the uniaxial compressive stress-strain curves of Al/SiC/Al_2O_3 hybrid nanocomposites at room temperature, and Table 3 summarizes the mechanical behavior based on these curves. Fabricated Al/2SiC/4Al_2O_3 hybrid nanocomposite was found to have a 0.2% offset compressive yield strength (CYS) of 113 ± 4 MPa and an ultimate compression strength (UCS) of 364.3 ± 3 MPa with a uniform strain of 76%. Further, with the addition of alumina (6 wt%), a decrease of UCS ~ 351 ± 4 MPa was observed for Al/2SiC/6Al_2O_3 hybrid nanocomposite.

Al/SiC/Al_2O_3 hybrid nanocomposites improved mechanical performance depending on several factors: (i) the presence of hard and uniform distributions of nanoreinforcements in the aluminum matrix and (ii) dispersion hardening effect and load-bearing capacity. However, the high amount of alumina causes a reduction in the aluminum matrix's hardness, which in turn causes a reduction in the bonding effect between the matrix and reinforcements.

Table 3 Mechanical behavior of Al/SiC/Al_2O_3 hybrid nanocomposites

Composition	Microhardness	Compressive properties		
	(Hv)	CYS (MPa)	UCS (MPa)	Failure strain (%)
Pure Al	40 ± 3	73 ± 5	305 ± 6	< 76
Al-2SiC	63 ± 2	85 ± 4	328 ± 4	< 76
Al-2SiC-2Al_2O_3	74 ± 1	94 ± 3	339 ± 5	< 76
Al-2SiC-4Al_2O_3	86 ± 4	113 ± 4	364 ± 3	< 76
Al-2SiC-6Al_2O_3	80 ± 2	107 ± 2	351 ± 4	< 76

4 Conclusions

Powder metallurgy combined with microwave sintering was used to make Al-SiC-Al_2O_3 hybrid nanocomposites with a fixed amount of SiC (2 wt%) and different amounts of Al_2O_3 (2, 4, and 6 wt%). We studied their microstructure, mechanical properties, and strengthening mechanisms. The following are the investigation's key findings:

- SEM micrographs revealed a uniform distribution of Al_2O_3 nanoparticles within the Al-SiC matrix.
- Microhardness increases from 40 ± 3 Hv for pure Al to 86 ± 4 Hv for Al-2SiC-4Al_2O_3 composite.
- The highest yield and compressive strength, 113 ± 4 and 364 ± 3 MPa, were obtained for the composite with 4 wt% of Al_2O_3, respectively.
- Attaining the uniform dispersion of dense ceramic nanoparticles (SiC and Al_2O_3) and their presence enhances

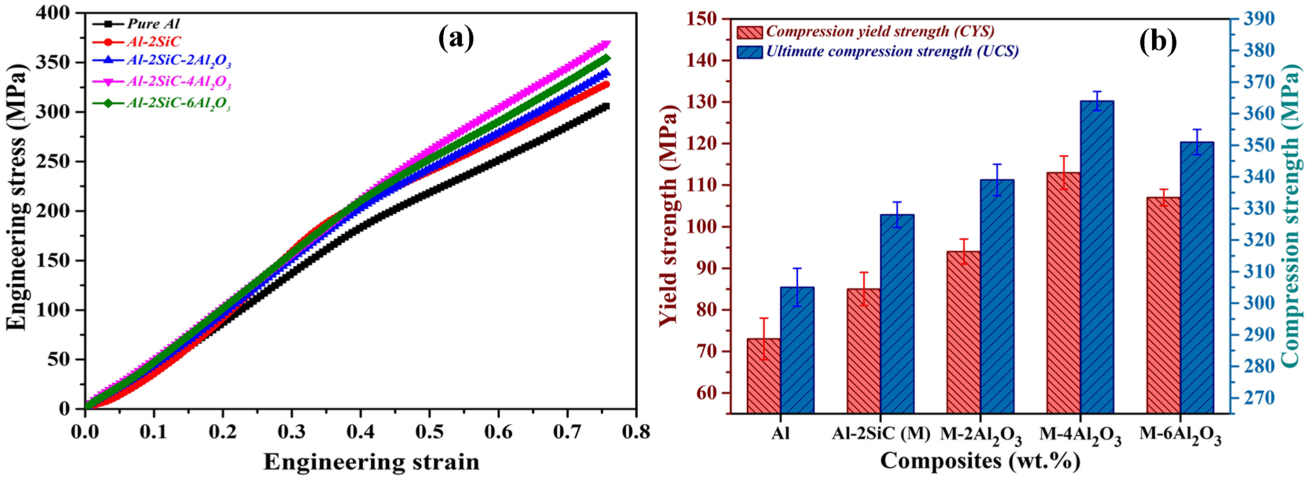

Fig. 7 **a** Engineering stress-strain curves and **b** corresponding compression data of the Al/SiC/Al_2O_3 hybrid nanocomposites

the mechanical behavior, activating the dispersion hardening phenomenon.
- Lightweight Al-based hybrid nanocomposite materials with superior mechanical properties are produced via a relatively low-cost microwave sintering method.

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Author contribution PRM, GM: conceptualization, methodology, software, writing—original draft preparation. RA, SSRL: data curation, visualization, investigation. RAS: supervision. AA: software, validation. AMAM, RGK: reviewing and editing.

Data availability Data can be made available upon request to the authors.

Declarations

Conflict of interest The authors declare no competing interests.

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