Exploring the microstructural and mechanical response of aluminum composites reinforced with SiN: a novel hybrid microwave sintering approach

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Abstract. This study investigates the microstructural and mechanical behavior of AA7075 aluminum matrix composites reinforced with varying weight percentages (1–8 wt%) of silicon nitride (SiN), processed via high-energy ball milling and hybrid microwave sintering. Microstructural characterization revealed uniform SiN dispersion and strong matrix–particle interfaces up to 6 wt%, while higher loadings led to agglomeration. Mechanical testing demonstrated significant improvements in tensile strength (149 MPa), compressive strength (237 MPa), and hardness (107 HV) at 6 wt% SiN, attributed to Orowan looping, load transfer, grain refinement, and Zener pinning mechanisms. Beyond this threshold, reinforcement agglomeration adversely affected performance. The results confirm that hybrid microwave sintering is a viable and energy-efficient route for producing high-strength aluminum-based composites, with 6 wt% SiN identified as the optimum reinforcement level.

1 Introduction

Therefore, AMCs have attracted wide attention in structural and high-performance uses in aerospace, automotive, and defense applications because of their high Specific strength, corrosion resistance, and thermal conductivity [1]. Among the different aluminum alloys, AA7075 is one of the strongest commercially available alloys and is commonly used in load-bearing aerospace applications. However, the addition of ceramic reinforcements can produce substantial mechanical and wear property improvements in this alloy, which has been used in the development of advanced AMCs for extreme service environments [2].

The incorporation of various ceramic reinforcements like SiC, Al_2O_3, B_4C, and Si_3N_4 into aluminum matrices has shown promising potential in enhancing hardness, strength, and high-temperature stability of these materials. Among these, Si_3N_4 is highly attractive because of its high hardness, superior thermal shock resistance, chemical inertness, and low coefficient

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of thermal expansion, as well as its ability to act as an effective reinforcement for high-performance AMCs. According to Dhanesh et al. [3], the addition of nano-Si_3N_4 to AA6061 resulted in a marked enhancement in tensile strength and thermal stability owing to grain refinement and particle strengthening effects. Besides, Sivasankaran et al. [4] have also reported improved wear resistance and microhardness in Al–Si_3N_4 composites prepared by traditional powder metallurgy technique.

Nevertheless, owing to density mismatch, poor wettability, and limited diffusion at the particle-matrix interface, attaining a proper dispersion of Si_3N_4 and an excellent interfacial bonding with aluminum remains a challenge. High-energy ball milling has been one of the most common treatments for enhancing distribution and improving matrix-particle interaction, as Lim et al. [5], in their study on Al-SiC composites, reported that ball milling resulted in enhancements in homogeneity and densification.

Sintering is another key element that determines the final microstructure and properties of powder-based composites. There are several disadvantages associated with conventional sintering methods, including long processing time, non-uniform grain growth, and lower energy efficiency. The advantages of microwave sintering include volumetric heating, rapid densification, and energy economy. Although the base material aluminum is found to be generally transparent to microwaves owing to its high reflectivity, hybrid microwave sintering, in which ceramic reinforcements like Si_3N_4 serve as microwave absorbers or susceptors, has successfully been used to effectively sinter such an aluminum-based system. Studies by Mishra and Sharma [6] and Li et al. [7] reported that microwave sintering significantly improved densification and grain boundary bonding in metal–ceramic systems over the conventional routes.

Recent publications, such as those by Venkateswara et al. and Manohar et al., focused on hybrid microwave-sintered Al composites with B_4C, ZrC, and TiC reinforcement, reporting significant enhancement in hardness and compressive strength due to refined microstructures and strong particle–matrix interfaces. In contrast, there is very little research on microwave sintering of AA7075/ Si_3N_4, especially using high-energy ball-milled powders, which forms the novelty of the present research. Hybrid microwave sintering offers several advantages compared with conventional sintering routes like resistance heating and furnace sintering. Conventional sintering uses only external heating, and this leads to longer processing times, heterogeneous temperature distribution, excessive grain growth, and increased energy input. In contrast, volumetric heating is facilitated by hybrid microwave sintering, where ceramic reinforcements like Si_3N_4 serve as highly effective microwave susceptors (absorbers) and thus facilitate rapid and uniform densification in otherwise microwave-transparent aluminum systems. At the same time, this approach results in reduced sintering cycle time and energy consumption, while improving matrix–reinforcement interfacial bonding and suppressing abnormal grain coarsening. Microwave sintering has indeed been successfully applied to reinforcements such as B_4C, ZrC, and TiC; however, AA7075/ Si_3N_4 remains almost unexplored, especially when combined with high-energy ball milling for improving dispersion [8]. This forms the key novelty of the present work.

While some previous studies have investigated AA7075–Si_3N_4 composites using conventional powder metallurgy or furnace sintering, their improvements in strength and hardness have generally been modest due to limited particle dispersion, non-uniform densification, and prolonged thermal exposure that promoted grain coarsening. In contrast, the present study combines high-energy ball milling (4 h milling at 250 rpm, BPR 10:1, with acetone as PCA) with hybrid microwave sintering (550 °C peak temperature, 30 min hold, N_2 atmosphere). This approach offers two key benefits: (i) homogeneous distribution of particles and refinement in grain structures through ball milling, and (ii) hybrid microwave sintering which ensures fast, volumetric heating that inhibits abnormal grain growth and improves matrix-reinforcement interfacial bonding. In addition, compared to previous studies

with limited, moderate improvements, this research achieved, at the optimum reinforcement level, a 57% improvement in tensile strength, 35% in compressive strength, and 35% in hardness with the addition of 6 wt% Si_3N_4, which unmistakably proves the superiority of the processing strategy adopted in [9].

In the present investigation, AA7075–SiN composites were prepared using a high-energy ball milling process followed by hybrid microwave sintering with the objective of studying the microstructural evolution and mechanical response. This work focuses on the following: uniform distribution of Si_3N_4 reinforcement in the aluminum matrix, optimization of microwave sintering conditions for maximum densification, and evaluation of mechanical properties such as tensile strength, hardness, and compressive behavior.

2 Materials and methods

2.1 Raw materials

The matrix material used in this study was gas-atomized AA7075 aluminum alloy powder (supplied by Nanoshell, India), characterized by an irregular morphology with particle sizes ranging from 15 to 45 μm, as confirmed by SEM analysis. The powder exhibited ≥99.7% purity, with its chemical composition shown in Table 1. Silicon nitride (SiN, NanoShell, India) was selected as the reinforcement, consisting of angular particles with diameters of 2–5 μm and a purity of ≥99.9%, predominantly in the α-phase.

Table 1. Chemical composition of AA7075 aluminium alloy (wt.%).

Element	Zn	Mg	Cu	Fe	Si	Mn	Cr	Ti	Al
% Weight	5.1–6.1	2.1–2.9	1.2–2.0	≤0.5	≤0.4	≤0.3	0.18–0.28	≤0.2	Balance

2.2 Composite powders preparation

To ensure homogeneous distribution of SiN within the AA7075 matrix, high-energy ball milling was performed using a Fritsch Pulverisette 5 planetary mill (Fritsch GmbH, Germany). The process employed tungsten carbide (WC) balls (Ø 3 mm) at a ball-to-powder ratio (BPR) of 10:1 and a rotational speed of 250 rpm for 4 hours, with intermittent pauses (15 minutes every 30 minutes) under an argon atmosphere to prevent oxidation-induced degradation. Acetone was used as Process Control Agent (PCA) in order to reduce the cold-welding phenomenon [10].

2.3 Compaction and sintering

The blended composite powders were then uniaxially compacted at 400 MPa using a D2 tool steel die (hardened to 60 HRC) into two geometries: rectangular-shaped specimens and then machined according to ASTM E8M standards through Wire cut EDM and cylindrical compression samples (Ø 10 mm × 15 mm, ASTM E9). Sintering was carried out in an industrial microwave furnace operating at 2.45 GHz in multi-cavity mode to ensure uniform heating. The optimized sintering profile included a heating rate of 10°C/min to a peak temperature of 550°C, held for 30 minutes under a nitrogen atmosphere (99.995% purity). Post-sintering, the composites were mechanically tested using an Instron 5982 universal testing machine (UTM) at a strain rate of 1 mm/min for both tensile (ASTM E8M) and

compression (ASTM E9) evaluations. Vickers microhardness measurements were conducted with a 500 gf load and 15-second dwell time.

2.4 Characterization of composites

For microstructural characterization, sintered samples were metallographically polished using sequential SiC emery papers (400–2000 grit) followed by a 1 μm diamond suspension. Etching was performed with Keller's reagent (2 mL HF + 3 mL HCl + 5 mL HNO_3 + 190 mL H_2O) for 10–15 seconds to reveal grain boundaries and reinforcement dispersion. Microstructural features, including SiN distribution, porosity, and interfacial bonding, were analyzed using scanning electron microscopy and X-ray diffraction (XRD, Cu-Kα radiation) for phase identification.

3 Results and discussions

Figure 1. shows BSE micrographs of AA7075–SiN composites at different reinforcement contents and highlights the morphological features, dispersion characteristics, and interfacial behavior of silicon nitride particles in the matrix. Figure 1(a), the microstructure of pure AA7075 alloy, consists of a reasonably homogeneous matrix with no second-phase reinforcements. Such a morphology is typical of sintered aluminum alloys, with fine grain boundaries and some residual porosity. This represents a basis for comparison against the reinforced composites. With the addition of 6 wt% SiN, Figure 1(b) demonstrates a uniform and fine dispersion of reinforcement particles throughout the matrix. The particles demonstrate good bonding with the surrounding aluminum matrix with evidence of appropriate interfacial integrity. The contact between the SiN particles and the matrix is intimate, suggesting that successful load transfer pathways are likely to be available with the effectiveness of Orowan strengthening, load-bearing reinforcement, and grain refinement mechanisms [11]. The hybrid microwave sintering approach appears to have provided a reasonably uniform thermal profile that has hindered localized melting or segregation and facilitated good interparticle diffusion.

At 7 wt% SiN, Figure 1(c) exhibits localized agglomeration of particles. Although the general dispersion is still acceptable, the clustered reinforcement may act as a stress concentrator and cause localized debonding or microcrack initiation upon mechanical loading. In spite of the development of such agglomerates, it is clear from the micrographs that these clusters are still well bonded to the matrix, implying coherent or semi-coherent interfaces. It is suggested that the interfacial strength is not significantly degraded, even for the elevated reinforcement level. This is more evident in Figure 1(d), showing a high-magnification (1000×) image of a reinforcement agglomerate in the 8 wt% composite. The SiN particles appear to be fused and embedded in the aluminum matrix without interfacial voids or gaps. Strong bonding at the matrix–reinforcement interface, even within an agglomerated zone, may still be favorable for strength up to a threshold. However, excessive agglomeration can prevent proper distribution of the applied stresses, limiting the strengthening effect [12].

Figure 2(a) illustrates the effect of varying SiN content on the tensile strength of AA7075 composites. For the unreinforced alloy, the tensile strength had a base value of 95 MPa and increased steadily with increasing SiN addition, reaching a peak of 149 MPa at 6 wt% reinforcement. This increase in strength by 57% may be understood in terms of several concurrent strengthening mechanisms: at low concentrations of SiN (1–4 wt%), the finely dispersed ceramic particles, as in Figure 1(b), serve as efficient barriers to dislocation motion resulting in Orowan looping, whereby dislocations are forced to bypass non-shearable particles. This increases the dislocation density and enhances yield strength. Additionally, the

reinforcement particles restrict grain boundary mobility during sintering, invoking the Zener pinning effect, which supports the retention of a refined grain structure and enhances the Hall–Petch strengthening response [13].

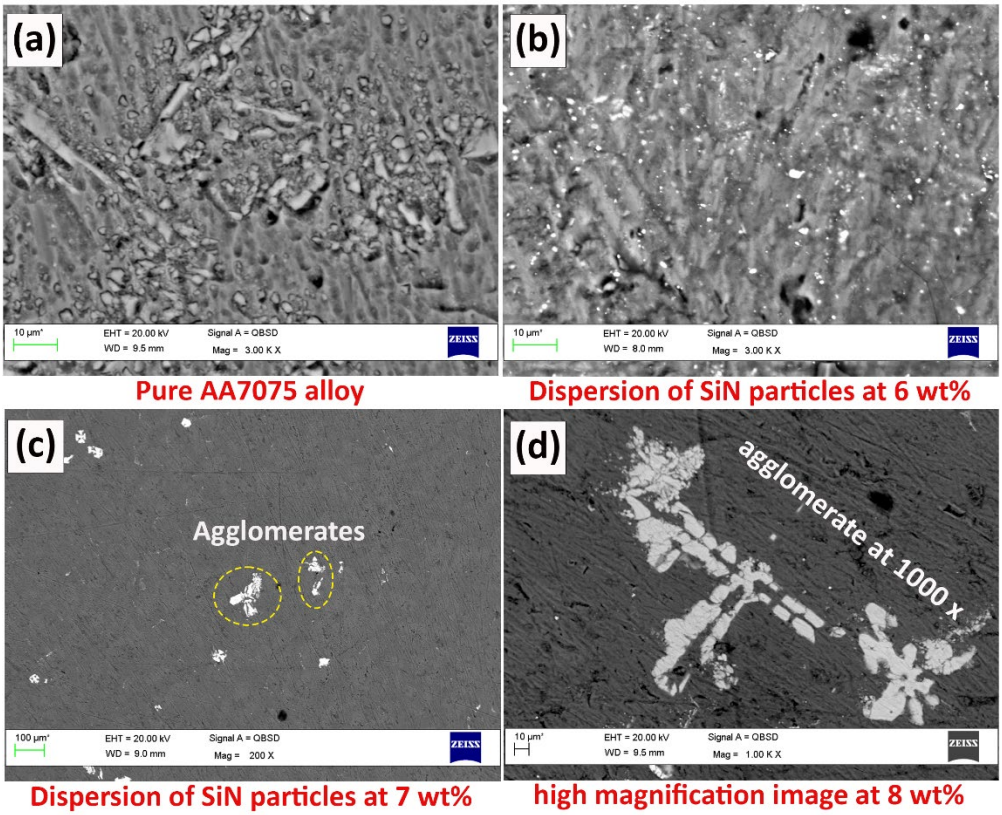

Fig. 1. SEM images of AA7075/SiN composite showing (a) Pure AA7075 alloy (b) Uniform dispersion of SiN at 6 wt% (c) Agglomerates of SiN at 7 wt% , and (d) High magnification of agglomerate shown in (c).

The most significant gain in tensile strength occurs between 4 wt% and 6 wt%, where the combined effects of load transfer, grain refinement, and optimal dispersion of SiN particles promote superior stress distribution and strain hardening. The strong particle–matrix interfaces observed in Figure 1(b) further enable efficient load transfer, where the stiff ceramic particles bear a portion of the applied load, thereby reducing the matrix stress concentration. However, a decline in tensile strength is observed beyond 6 wt%, with values dropping to 141 MPa at 7 wt% and 125 MPa at 8 wt%, respectively. This performance degradation is directly related to the particle agglomeration observed in Figure 1(c) and 1(d), which forms clusters of SiN, reducing the effective reinforcement surface area and generating zones of stress concentration from which microcracks initiate under tensile loading.

The same enhancement trend is observed in Figure 2(b) for compressive strength. In this respect, the compressive strength increased monotonically from its unreinforced AA7075 value of 175 MPa to a maximum of 237 MPa at 6 wt% SiN with an increase of about 35%. This increase is therefore due to the mutual effect of the refined microstructure, dense sintered matrix, and homogeneously distributed reinforcement promoting improved resistance to plastic deformation of the material under compressive load. The hybrid microwave sintering technique played a significant role in preserving fine grain size due to the rapid volumetric

heating, which restricted grain growth due to uniform heat distribution. At the same time, the Zener pinning mechanism [14], through which reinforcement particles anchor the grain boundary during sintering also plays a major role in such densification and microstructural stability. However, the compressive strength was reduced to 225 MPa and 208 MPa at 7 wt% and 8 wt%, respectively. The observed compromise in structural integrity provided by reinforced agglomerates, which promotes localized deformation, is once again responsible for this trend.

Figure 2(c) shows that in terms of surface hardness, there is almost a linear enhancement with the increase in SiN content. Hardness increased from 79 HV for pure AA7075 to 107 HV at 8 wt%, which amounts to an increase of 35%. Unlike the tensile and compressive strengths, hardness continues to improve at higher SiN contents since it is influenced only by the local surface resistance to indentation, which benefits from the increased availability of hard ceramic particulates irrespective of agglomeration. The high hardness of SiN itself (~15–20 GPa) contributes directly to the dispersion strengthening effect, where the embedded particles resist localized plastic flow, thereby constraining matrix deformation. Further, the additional dislocation density around these hard particles, arising due to thermally induced mismatch dislocations and Orowan looping, also provides extra resistance to penetration [15].

The progressive agglomeration of SiN observed at loadings above 6 wt% is attributed to both intrinsic particle interactions and processing limitations. As reinforcement fraction increases, the mean inter-particle spacing reduces considerably and particle–particle interactions (van der Waals forces and mechanical interlocking) begin to dominate over particle–matrix interactions, promoting cluster formation. The high specific surface area of the micron- and sub-micron-sized SiN particles increases their surface energy, rendering them thermodynamically prone to agglomeration unless sufficiently strong deagglomeration energy or dispersing agents are applied during powder blending. Processing-related causes also play a major role. During high-energy ball milling, insufficient milling energy, sub-optimal ball-to-powder ratio, or short milling duration can leave hard-to-break agglomerates intact; conversely, excessive cold welding of ductile AA7075 particles onto SiN surfaces can cause adhesive clustering. During compaction and green-body handling, differential particle packing and local particle segregation can produce reinforcement-rich zones which densify differently during sintering. During hybrid microwave sintering, local volumetric heating of SiN-rich regions (which act as microwave susceptors) may accelerate local necking/partial bonding between neighbouring SiN particles or between SiN clusters and the matrix, thereby stabilizing agglomerates rather than dispersing them. Differential thermal expansion between Al and SiN during heating/cooling can also generate local stresses that promote particle clustering or microcrack initiation at agglomerate sites.

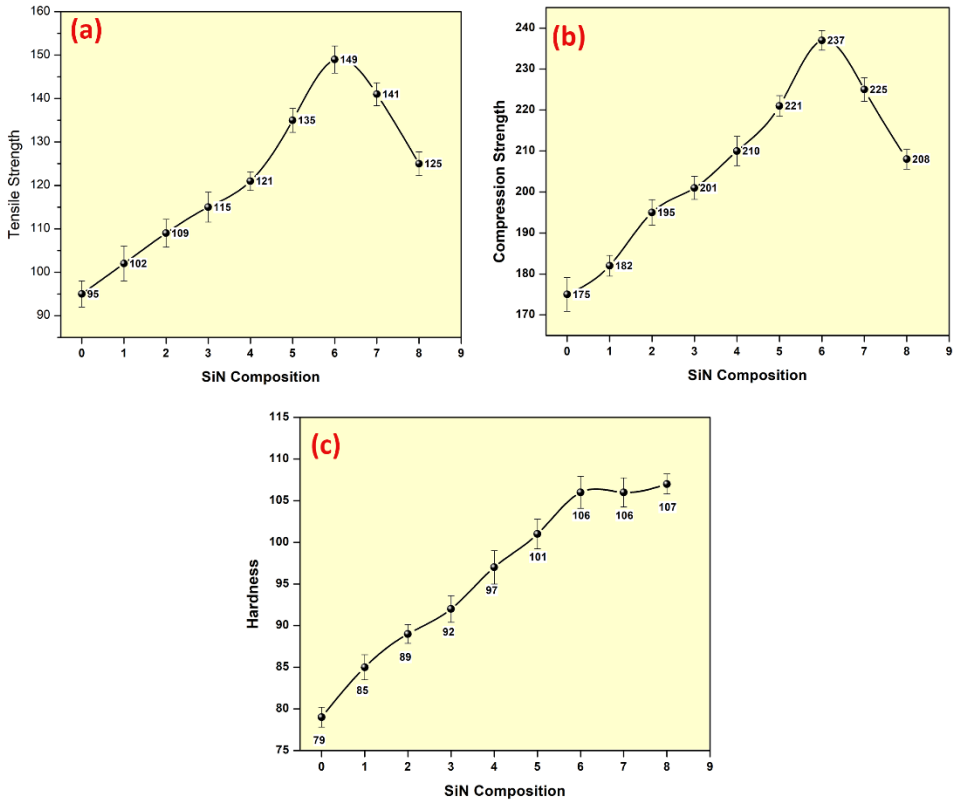

Fig. 2. Mechanical properties of AA7075/SiN composite showing (a) Tensile strength (b) compression strength, and (c) Hardness.

The correlation between the mechanical properties and microstructure is thus strongly evident. Up to 6 wt%, the AA7075–SiN composites exhibit a synergistic balance of uniform dispersion, strong interfaces, and refined microstructure, resulting in improved tensile, compressive, and hardness values. The hybrid microwave sintering with high-energy ball milling ensures homogeneity in the particle distribution and, simultaneously, improves the sinterability, which is a key aspect of obtaining strong and reliable composite systems. However, beyond an optimal reinforcement level, the initiation of agglomeration in the particles introduces heterogeneity and stress concentrators into the material, despite the presence of remaining strong interfacial bonds. Therefore, this study establishes 6 wt% SiN as the optimal reinforcement level for achieving improved mechanical properties with no microstructural compromise.

While the present work delineates the feasibility of hybrid microwave sintering during the processing of AA7075–SiN composites, a few issues are raised. Firstly, scaling up to industrial component sizes is quite a challenge owing to the complexity in achieving uniform microwave penetration and resultant heating for large or intricate geometries. Secondly, though it is expected to be economically feasible based on energy savings and reduced cycle times, in contrast, industrial microwave facilities have a higher initial investment cost compared with conventional resistance furnaces. Third, the localized overheating of SiN-rich zones could potentially result in thermal stresses and/or undesirable interfacial reactions, which necessitates a careful optimization of sintering parameters with respect to the possibility of phase transformations or property degradation.

Therefore, future studies need to concentrate on the scaling of the process for larger components and the validation of consistent performance; long-term durability and thermal stability tests of hybrid microwave-sintered composites; integrating advanced process modeling together with in-situ temperature monitoring to predict and control the microstructural evolution; and benchmarking lifecycle costs of the microwave and conventional sintering routes toward their industrial adoption. Efforts in this direction will definitely strengthen the path required for transition of hybrid microwave-sintered aluminum composites from laboratory-scale feasibility to large-scale engineering applications.

4 Conclusion

In the present work, AA7075 aluminum matrix composites with different weight percentages (1–8 wt%) of SiN have been successfully synthesized by combining high-energy ball milling and hybrid microwave sintering. The microstructural features and mechanical performance of the composites with varying SiN content have been studied in detail. Microstructural studies showed a homogeneous dispersion up to 6 wt% SiN, with good interfacial bonding between reinforcement and matrix. Agglomeration of particles was observed at higher reinforcement levels (7–8 wt%) that, though disrupting the homogeneity of the composite locally, still ensured reasonably good bonding at the matrix–reinforcement interface. The microwave sintering route suppressed grain growth and enhanced densification owing to the rapid and volumetric heating, while ball milling ensured a uniform distribution of particles ahead of sintering.

The improvements in tensile strength, compressive strength, and hardness were strongly dependent on SiN content, with the maximum values occurring at 6 wt% reinforcement. At this optimum reinforcement level, tensile strength increased by 57% (from 95 MPa to 149 MPa), compressive strength by 35% (from 175 MPa to 237 MPa), and hardness by 35% (from 79 HV to 107 HV), relative to the unreinforced alloy. Such improvements were related to a conjunction of Orowan strengthening, load transfer, grain refinement (Hall–Petch effect), and Zener pinning, which were made possible through hybrid microwave sintering and homogeneous distribution of reinforcements. Beyond 6 wt% SiN, a slight decrease in tensile and compressive strengths was recorded due to the agglomeration of particles, which acted as a stress concentrator and reduced the effectiveness of both the load transfer and dispersion strengthening mechanisms.

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