

Article

Microstructure, Mechanical and Ignition Characteristics of Si_3N_4 Reinforced Magnesium Matrix Nanocomposites

Mahammod Babar Pasha ¹, Rajamalla Narasimha Rao ^{1,*}, Syed Ismail ¹ and Manoj Gupta ^{2,*} 

¹ Department of Mechanical Engineering, National Institute of Technology Warangal, Warangal 506004, India; pashamd@student.nitw.ac.in (M.B.P.); syedismail7@nitw.ac.in (S.I.)

² Department of Mechanical Engineering, National University of Singapore, 9 Engineering Drive 1, Singapore 117576, Singapore

* Correspondence: rnrao@nitw.ac.in (R.N.R.); mpegm@nus.edu.sg (M.G.); Tel.: +91-8639772117 (R.N.R.); +65-6516-6358 (M.G.)

Abstract: Lightweight magnesium-based materials have received attention in the automobile sector as a solution to minimize fuel consumption and greenhouse gas emissions. Magnesium has great weight-reduction potential in the aerospace sector, but its low ignition temperature limits its utilization. Improving magnesium's ignition resistance is critical for aerospace applications. The present study developed Mg/ Si_3N_4 nanocomposites to improve the ignition resistance to address this limitation. The nanocomposites were prepared by ultrasonically-assisted stir casting with 0.5, 1, and 1.5 vol% Si_3N_4 nanoparticles. The effect of Si_3N_4 nanoparticles on the ignition and compression characteristics was examined. SEM micrographs showed the homogeneous dispersion of Si_3N_4 nanoparticles with negligible clustering. Notably, the nanocomposites' ignition resistance was increased by increasing the vol% of the Si_3N_4 nanoparticles. Adding 1.5 vol% Si_3N_4 nanoparticles resulted in the highest ignition temperature of 614 °C, 34 °C higher than pure magnesium. Similarly, the compressive properties were enhanced with the progressive addition of Si_3N_4 nanoparticles. The inclusion of 1.5 vol% Si_3N_4 nanoparticles resulted in a maximum compressive yield strength of 118 MPa and ultimate compressive strength of 323 MPa.

Keywords: magnesium nanocomposites; silicon nitride (Si_3N_4) nanoparticles; ultrasonic stir casting; ignition temperature; compressive strength



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1. Introduction

Over the past few decades, rising fuel prices and strict regulations on CO₂ emissions have led the transportation sector to strive for lightweight and robust materials [1–3]. Replacing traditional materials, such as high-strength steel and aluminum, with magnesium-based materials makes automobiles lighter, thus improving fuel utility [4]. Magnesium is ~33% lighter but has a strength close to aluminum, making it a promising substitute for aluminum in lightweight structural components. In addition to magnesium's low density, high specific strength, castability, machinability, recyclability, thermal stability, damping capacity, and electrical conductivity make magnesium an excellent choice in the automobile and electronic sector [5]. Moreover, magnesium-based materials have the essential scope for use in the aerospace industry [6,7]. Using magnesium in aircraft seats, seat tracks, overhead bins, and doors will significantly reduce aircraft weight. Inside the aircraft cabin, it is estimated that the mass distribution of the seats is 42–44% of aluminum, 9.5–15% of plastics and 7.8–10% of steel [8,9]; replacing these with magnesium will significantly affect the overall performance. However, magnesium-based materials are still not employed in these applications due to the previously imposed ban on the use of magnesium in aircraft due to its high affinity for oxygen (i.e., low oxidation resistance) [10,11].

The main disadvantages of this low oxidation resistance are easy ignition when in contact with an adequate electric spark or flame and the inability to self-extinguish [12–14].

Despite being alloyed, magnesium is an unsuitable material to extinguish using the current extinguishers in aeroplanes. An effective way to improve the ignition resistance of magnesium is by enhancing its oxidation kinetics by adding nano reinforcement particles into the magnesium matrix [15–17]. The incorporation of nanoparticles into the magnesium matrix considerably improves the ignition temperature by forming a stable and more protective oxide layer on magnesium's surface. Furthermore, the low thermal conductivity of the nanoparticles compared to magnesium reduces the thermal conductivity of the composites and improves thermal stability and ignition resistance [17,18].

Recent studies have analyzed the influence of various nano reinforcement particles on the ignition resistance of the magnesium-based materials. The addition of 10 wt% silicon carbide micron particles [19] to the AM60B alloy increased the ignition temperature to 603 °C from 525 °C. Adding 2 vol% silica nanoparticles (SiO_2) [20] into the magnesium matrix improved the ignition resistance by 30 °C. The inclusion of SiO_2 nanoparticles delayed the onset of ignition by developing a stable and protective oxide film on the surface. Similarly, adding 1.5 vol% of samarium oxide (Sm_2O_3) nanoparticles [21] to the magnesium matrix improved the ignition resistance. The addition of nano-ceria (CeO_2) nanoparticles [22] to the pure magnesium matrix improved the ignition resistance and compression strength by 21% and 32% more than pure Mg. The addition of CeO_2 nanoparticles resulted in a stable and protective CeO_2 rich oxide layer, with a Pilling–Bedworth ratio (PBR) of 1.07 (oxide layer with a PBR value greater than 1 and less than 2 is stable and protective [18]), causing the delayed ignition of the composites. Similar to this, the addition of 25 wt% of hollow glass microballoons [23] into the magnesium matrix improved the ignition resistance by developing a more protective oxide film, with a PBR value of 1.89. The addition of 1.5 vol% calcium oxide (CaO) nanoparticles to the magnesium matrix significantly improved the ignition resistance to 831 °C, which is 33% higher than the magnesium. These promising results of the particle-reinforced magnesium nanocomposites provide significant motivation for researching novel reinforcement particles to develop new magnesium-based composites. Furthermore, with the Federal Aviation Administration (FAA) lifting the Mg restriction in 2015, there is increasing attention in substituting aluminium in the aerospace sector with magnesium [24].

Silicon nitride (Si_3N_4) is a non-oxide ceramic material with a density of 3.4 g/cc, with high thermal conductivity (30 W/m.K), low thermal expansion coefficient ($3 \times 10^{-6}/^\circ\text{C}$), and high compressive strength (4500 MPa) [25]. In addition, its high-temperature stability, high fracture toughness, and high thermal shock resistance make it an excellent choice of reinforcement particle for improving the ignition and compression strength of magnesium. However, no study is available in the open literature on the improvement of ignition resistance and compressive response of magnesium by reinforcing it with Si_3N_4 nanoparticles using the ultrasonic-assisted stir casting technique.

Accordingly, in the present work, Mg/ Si_3N_4 nanocomposites were fabricated through the ultrasonic-assisted stir casting method by varying the nanoparticles' amount (0.5, 1 and 1.5 vol%). Furthermore, the effect of the nanoparticles on the ignition and compression properties was thoroughly investigated.

2. Materials and Methods

Mg/ Si_3N_4 nanocomposites, by varying the volume percentage (0%, 0.5%, 1% and 1.5 vol%) of Si_3N_4 nanoparticles in the pure Mg matrix, were fabricated using the ultrasonic-assisted stir casting method. The ingots of magnesium were measured out beforehand, and then they were melted in a resistance heating furnace at 750 °C. Once a homogenous melt had been achieved, preheated Si_3N_4 nanoparticles were added, and a mechanical stirrer was used to mix the composite mixture for 10 min. After this, ultra-sonication was carried out for 5 min, for the uniform distribution of the nanoparticles. Finally, the molten material was transferred into a steel mould.

The wire cut samples were polished and etched to investigate the Si_3N_4 particle distribution in the Mg matrix using a scanning electron microscope, equipped with energy-

dispersive spectroscopy (Vega LMU 3, TESCAN, Brno, Czechia). An X-ray diffractometer was used to perform the phase analysis of the samples. The experimental density was determined using Archimedes principle and the theoretical density using the rule of mixtures. The ignition temperature of the samples was evaluated using a thermogravimetric analyzer (TGA/DSC3+, Mettler Toledo, Mumbai, India). Three samples for each composition with the dimensions of $2 \times 2 \times 1 \text{ mm}^3$ were tested in an aluminium crucible from 30 to 750 °C, with a heating rate of 10 °C/min. Furthermore, the samples were subjected to compression testing in accordance with ASTM E9-09. A universal testing machine (model M-50, Fine Spray India, Bangalore, India) was used to test five samples for each composition with a length to diameter ratio of 1, at a strain rate of 8.33×10^{-5} .

3. Results and Discussion

3.1. Microstructural Characterization

Figure 1 shows SEM micrographs of pure Mg and the nanocomposites with a relatively uniform distribution of the Si_3N_4 nanoparticles (indicated with white arrows) across the Mg matrix. The distribution is more homogeneous in the 0.5 vol%, and 1 vol% Si_3N_4 particle-reinforced nanocomposites. The nanocomposites with 1.5 vol% Si_3N_4 nanoparticles showed near uniform distribution with some clustered nanoparticles (indicated with white circles). The observed uniform distribution is mostly due to the excellent ultra-sonication treatment used during nanocomposite fabrication. The ultrasonic vibrations in the melt dispersed the Si_3N_4 nanoparticles substantially. Furthermore, the appropriate stirring parameters and nanoparticle preheating increased the wettability between the Mg and nanoparticles. The EDX spectrum results of the Mg-1.5 Si_3N_4 nanocomposite (Figure 2) show the elemental composition of Mg, Si and N elements.

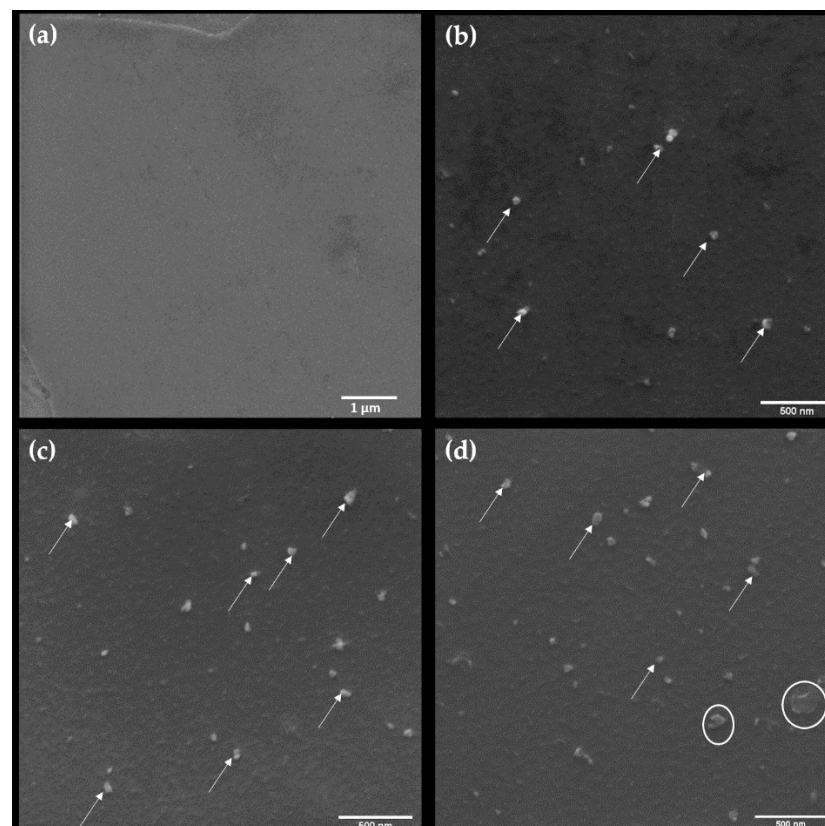


Figure 1. SEM images depicting the dispersion of nanoparticles in the (a) pure Mg, (b) Mg-0.5 Si_3N_4 , (c) Mg-1.0 Si_3N_4 , (d) Mg-1.5 Si_3N_4 .

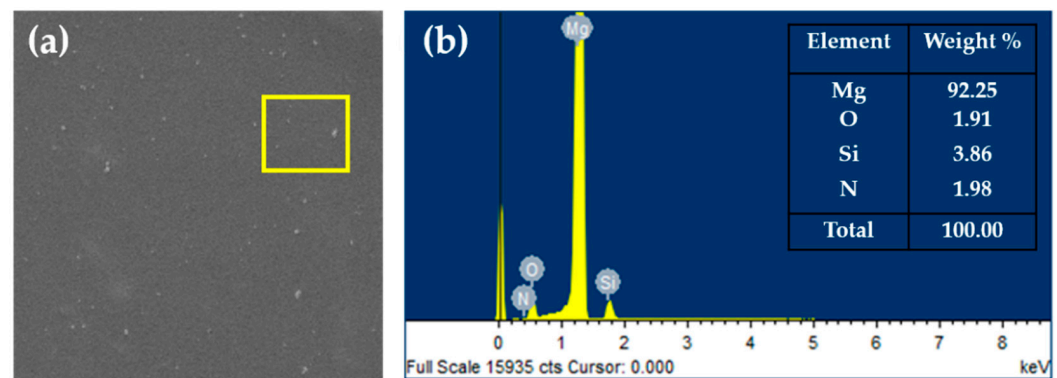


Figure 2. Representative SEM micrograph and EDX spectrum of the Mg-1.5Si₃N₄ nanocomposite. (a) SEM micrograph of mapped area, (b) EDX spectrum with elemental composition.

Figure 3 shows the XRD analysis results of the samples. The primary peaks corresponding to pure Mg were observed in all the samples. However, the peaks corresponding to Si₃N₄ were less intense in the XRD spectrum, due to a relatively low volume fraction (<2 vol%) of the particles in the matrix [26]. Nonetheless, the minor peak at 2θ of 70.7° corresponds to a plane at (2 0 2) in the nanocomposites, confirming the existence of Si₃N₄. Furthermore, there were no apparent peak shifts, suggesting that interfacial interaction was low throughout the casting process. The lack of reaction products and textural randomization caused by the inclusion of Si₃N₄ nanoparticles aid in the plastic deformation of the nanocomposites.

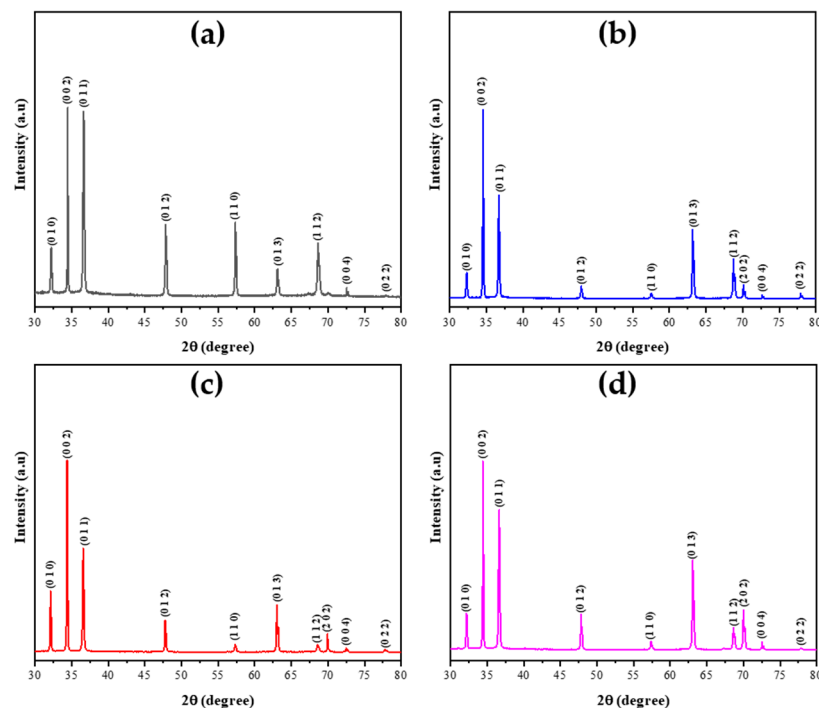


Figure 3. XRD analysis results of the samples: (a) pure Mg, (b) Mg-0.5Si₃N₄, (c) Mg-1.0Si₃N₄, and (d) Mg-1.5Si₃N₄.

3.2. Density and Porosity

Figure 4 shows the density and porosity values of the pure Mg and Mg/Si₃N₄ nanocomposites. The theoretical and experimental densities of the nanocomposites increased with Si₃N₄ vol%. This is due to the significant difference between the densities of the matrix and Si₃N₄ reinforcement. Among the nanocomposites, the 1.5 vol% Si₃N₄ nanocomposite has a maximum porosity of 1.3%. The comparatively high vol% of Si₃N₄

reinforcing particles in the Mg-1.5 Si₃N₄ nanocomposite leads to greater particle concentration zones, increasing the possibility of voids or pores. Pure Mg and the nanocomposites have porosity values below 1.5%, making them dense materials. Low porosity is due to the appropriate stirring conditions, ultra-sonication, and limited nanoparticle agglomeration.

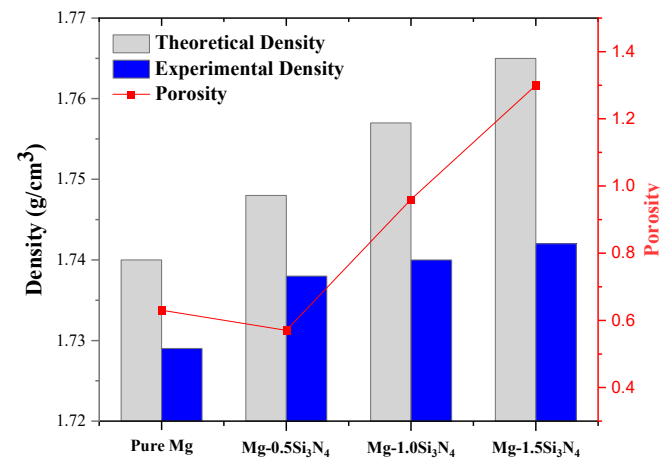


Figure 4. Density and porosity results of the samples.

3.3. Ignition Response

The temperature at which a sudden increase in the sample mass occurs was considered as the ignition temperature [27]. In the current study, the mass gain occurred from 575 °C to 750 °C due to considerable oxidation followed by ignition of the sample. The ignition temperature of the pure Mg increased by increasing the addition of Si₃N₄ nanoparticles. Adding 0.5 vol% of Si₃N₄ improved the ignition temperature to 596 °C, ~3% greater than pure Mg (580 °C). The further addition of 1 and 1.5 vol% Si₃N₄ improved the ignition temperature to 603 °C and 614 °C, which is ~4% and ~6% more than pure Mg. Figure 5 shows that the Mg/Si₃N₄ nanocomposites have greater ignition resistance than AZ-series (aluminium and zinc are constituent elements), ZK-series (zinc and zirconium are constituent elements), and AM-series (aluminium is the major constituent element) magnesium alloys. The ignition temperature of the Mg-1.5Si₃N₄ nanocomposite is higher than all the traditionally used magnesium alloys.

The significant improvement in ignition temperature of the Mg/Si₃N₄ nanocomposites can be attributed to the following phenomenon. The ignition phenomenon of pure Mg primarily depends on oxidation kinetics [8]. At temperatures below 450 °C, the oxide layer developed on the surface of Mg is reasonably protective and restricts subsequent oxidation. However, at temperatures more than 450 °C, the Gibbs free energy of exothermic oxide formation causes a rapid increase in local heat, leading to the metal melting beneath the oxide layer and generating cracks in the layer. Further heat raises the local temperature, resulting in vapour pressure in the entrapped liquid. This pressurized fluid explodes through the cracks and reacts with oxygen, resulting in the ignition of Mg [8]. In a sense, ignition only occurs when the protective oxide layer breaks and loses its ability to inhibit the reaction with the oxygen. The Pilling–Bedworth ratio (PBR) denotes the oxide layer’s quality formed on a metal surface. The oxide layer with a PBR value of less than 1 and more than 2 will provide poor protection. As MgO exhibits a PBR value of 0.81, the ignition is relatively easy to start. The addition of reactive elements, such as alloying elements and thermally stable nanoparticles, can aid in the development of a more stable and dense surface oxide layer, thereby improving Mg ignition resistance [18,28].

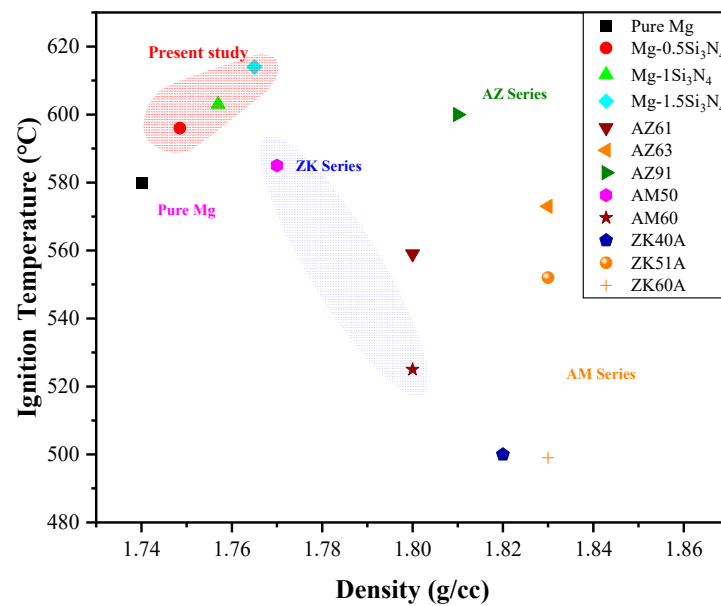


Figure 5. Ignition temperatures of widely available magnesium alloys [29], in comparison to the current study.

In the current work, the increased ignition resistance of the nanocomposites is attributed to the formation of a denser and more stable oxide layer below the thin surface layer of MgO. As the temperature increases, the thin Mg oxide layer breaks away, leaving behind a thick oxide layer, which further protects by restricting the reaction between Mg vapour and oxygen, thereby increasing the ignition resistance [30,31]. Additionally, the low thermal conductive Si_3N_4 nanoparticles act as insulating sites in the pure Mg matrix, which improves its thermal stability and ignition resistance. A correlation between the thermal conductivity and nanocomposite ignition temperature was established to better understand the influence of nanoparticles on ignition behaviour. Using the rule of mixtures, the theoretical thermal conductivity (K_c) of the nanocomposites was determined (refer to Equation (1)) by considering the thermal conductivity of base matrix (K_m) pure Mg and reinforcement (K_r) Si_3N_4 as 135 and 30 (W/m.K), respectively, for each volume percentage (V_c) of Si_3N_4 nanoparticles.

$$K_c = K_r V_c + (1 - V_c) K_m \quad (1)$$

The calculated thermal conductivity values are plotted (Figure 6) against the respective ignition temperatures of the samples. The ignition temperature of the Mg/ Si_3N_4 nanocomposites improved as thermal conductivity decreases because of the fact that the reinforced nanoparticles acted as insulation spots in the Mg matrix.

3.4. Compression Test

Table 1 shows the room-temperature compression test results. The variation of 0.2% compressive yield strength (0.2% CYS), the ultimate compressive strength (UCS), and failure strain are shown in Figure 7. Increasing the vol% of Si_3N_4 enhanced 0.2% CYS and UCS of magnesium. The 0.2% CYS of Mg increased from 63 MPa to 78 MPa, 101 MPa, and 118 MPa with the addition of 0.5, 1, and 1.5 vol% Si_3N_4 nanoparticles, respectively. Similarly, the UCS increased with the increasing addition of Si_3N_4 nanoparticles with 1.5 vol% Si_3N_4 nanocomposites and a maximum UCS of 323 MPa, which is 41% higher than the pure Mg. The failure strain (FS) of the nanocomposites increased up to the inclusion of 1 vol% Si_3N_4 nanoparticles, with a maximum FS of 21.2%, which is 30% greater than pure Mg. However, with the addition of 1.5 vol % Si_3N_4 nanoparticles, the FS value was reduced to 18.2%. Nevertheless, all of the nanocomposites had a higher failure strain than pure Mg.

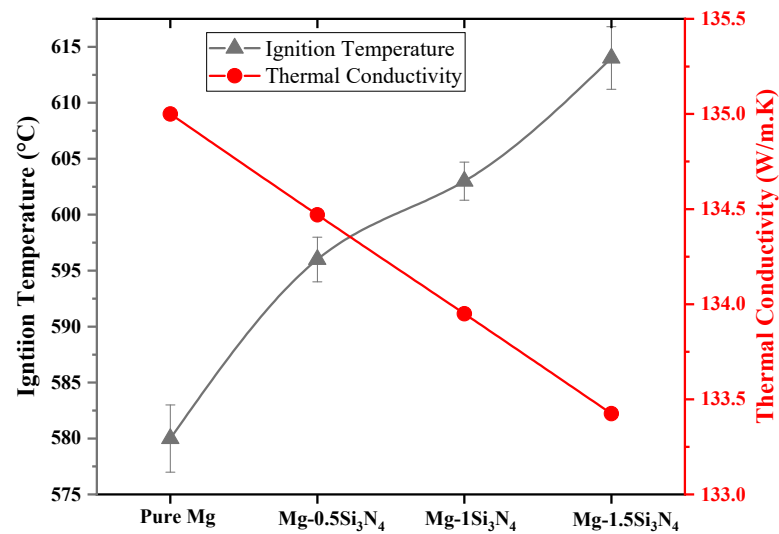


Figure 6. Thermal conductivity of pure Mg and Mg/Si₃N₄ nanocomposites as a function of ignition temperature.

Table 1. Compression test results of the samples.

Sample	0.2% CYS (MPa)	UCS (MPa)	Failure Strain (%)
Pure Mg	63 ± 5	229 ± 7	16.3 ± 0.5
Mg-0.5Si ₃ N ₄	78 ± 4	252 ± 9	18.7 ± 0.8
Mg-1.0Si ₃ N ₄	101 ± 6	296 ± 11	21.2 ± 0.6
Mg-1.5 Si ₃ N ₄	118 ± 8	323 ± 9	18.2 ± 0.8
Mg-0.4AlN [32]	72 ± 5	314 ± 20	17.5 ± 0.6
Mg-1.2BN [26]	109 ± 4	307 ± 6	17.6 ± 2
Mg-2.5TiO ₂ [33]	101 ± 9	305.5 ± 11	22 ± 2
Mg-2SiO ₂ [34]	89 ± 1	207 ± 3	21.87 ± 2.1
Mg-1.5CeO ₂ [22]	111 ± 2	329 ± 7	19.7 ± 0.8

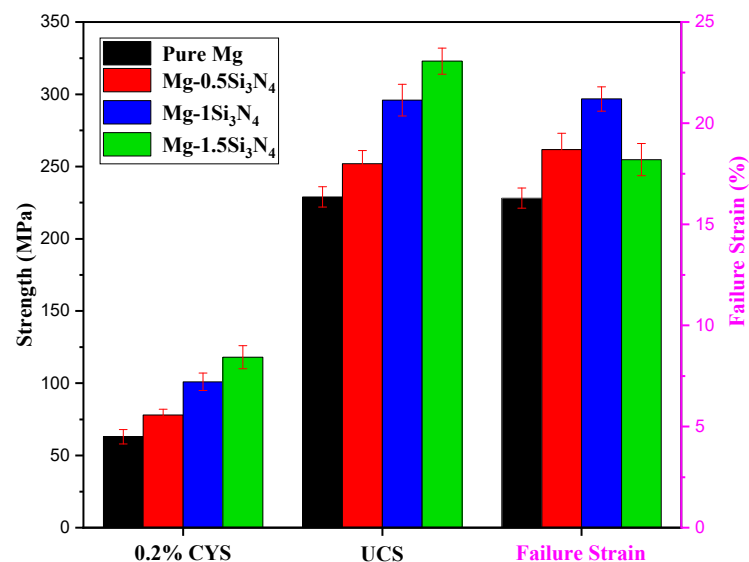


Figure 7. The effect of Si₃N₄ nanoparticles on compression properties.

The significant improvement in the compressive yield strength and ultimate strength of the Mg/Si₃N₄ nanocomposites may be due to the effective strengthening mechanisms, such as (i) the load bearing effect (efficient load transfer from soft matrix to hard Si₃N₄ nanoparticles due to better interfacial integrity), (ii) Orowan strengthening (caused from

the presence of Si_3N_4 nanoparticles), and (iii) dislocation strengthening (resulting from the increased dislocation density in the matrix and reinforcement interface due to the difference in the CTE and elastic modulus between the Mg matrix and Si_3N_4 nanoparticles).

4. Conclusions

Mg nanocomposites with Si_3N_4 as reinforcement were successfully fabricated using the ultrasonic-assisted stir casting method. The effect of Si_3N_4 nanoparticles on the Mg matrix's ignition temperature and compression properties was investigated. From this study, the following conclusions are drawn:

1. The ultrasonic vibration during the fabrication process resulted in a uniform distribution of Si_3N_4 nanoparticles in the Mg matrix. The distribution is more homogeneous in the 0.5 and 1 vol% Mg/ Si_3N_4 nanocomposites compared to the 1.5 vol% Si_3N_4 nanoparticles, as the latter had some clustered nanoparticles when observed under SEM;
2. Similar to the EDS analysis, the XRD results revealed the presence of Mg and Si_3N_4 . Moreover, the XRD analysis showed no evidence of interfacial reaction products;
3. The porosity of the samples increased with the increasing vol% of the Si_3N_4 nanoparticles, with Mg-1.5 Si_3N_4 showing a higher porosity (1.3%) compared to the remaining samples;
4. The ignition temperature of the nanocomposites is enhanced by increasing the amount of the Si_3N_4 nanoparticles by forming a dense and more stable oxide layer on the surface. The nanocomposite with 1.5 vol% Si_3N_4 nanoparticles showed the highest ignition temperature of 614 °C, ~6% more than pure Mg;
5. Adding Si_3N_4 nanoparticles gradually improved the compressive yield strength and ultimate strength, with 1.5 vol% Si_3N_4 nanocomposites having a maximum yield strength of 118 MPa and an ultimate strength of 323 MPa. The failure strain improved with the addition of Si_3N_4 nanoparticles, with 1 vol% Si_3N_4 nanocomposites displaying a maximum failure strain of 21.2%, ~30% greater than pure Mg.

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