

Physical, Mechanical and Morphological Characterization of A356/Si₃N₄ Nanoparticles Stir Casting Composites

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ABSTRACT

A356 alloy based composites are extensively used in different component industries like components of automobile parts owing to pronounced strength to weight ratio. In the current paper, A356/Si₃N₄ nanocomposites are fabricated by means of stir casting by varying Si₃N₄ reinforcement nanoparticles. Silicon Nitride (Si₃N₄) nanoparticle is intermixed to Al powder mechanically to develop their wettability among the particles of A356/Si₃N₄ nanocomposites. The Si₃N₄ nanoparticle is integrated by altering weight percentage. The electromechanical stirring process to produce the vortex is taken up to spread Si₃N₄ nanoparticles in the liquefied matrix dispensed into a permanent mould. Morphological investigation of the composite specimen is accomplished by TEM. Based on the study, it can be acquired that the strengthening by Si₃N₄ nanoparticles promotes the strength and hardness of the fabricated nanocomposites. The maximum tensile strength is depicted to be 319 MPa for A356/5%Si₃N₄ nanocomposites whereas hardness is increased from 43 HBN to 86 HBN. The physical

properties such as density and porosity are also increased due to the presence of Si_3N_4 nanoparticles. The maximum porosity of 1.12% was predicted at 5 wt. % of Si_3N_4 . The TEM examination discloses the presence of Si_3N_4 nanoparticles in the fabricated composites. Additionally, the current research guidance has ability to afford a monitor to the industrialized preparation of A356/ Si_3N_4 nanocomposites.

Keywords: Al-nanocomposites, Stir casting, Porosity, Hardness, Tensile strength, TEM.

INTRODUCTION

Light metal such as Al, Mg and Ti strengthened by means of several ceramic elements such as SiO_2 , SiC, WC, Al_2O_3 , Si_3N_4 , TiC, TiB_2 , BN, B_4C , TiO_2 , CN, Graphite, etc have low density with enhanced wear, corrosion, good thermal resistance, and mechanical properties which have been extensively studied by the research scientists (Kumar & Devaraju, 2021; Shettar et al., 2021; Zheng et al., 2020; Suresh et al., 2019; Selvam et al., 2018). They are used in a number of structural manufacturing sectors. Among the ceramic materials available for reinforcement, Silicon Nitride (Si_3N_4) ceramic is non-oxide having elevated melting temperature, high hardness, and lower density which make its uses in the various automotive sectors. Silicon Nitride (Si_3N_4) ceramic is appropriate for high loads, wear resistance, corrosion resistance, ball bearings, heat exchangers, turbine blades (Shoujiang et al., 2020; Parveen et al., 2019). Further, Si_3N_4 ceramic is widely used for a number of different parts of engine components (Kumar et al., 2016; Ramesh et al., 2010). Because of which Si_3N_4 ceramic particles are useful as a reinforcement for light alloys. Moreover, aluminium matrix composites (Al/ Si_3N_4) reinforced with Si_3N_4 particles have higher thermal contraction, extraordinary strength with respect to density, as well as better dimensional constancy (Wang et al., 2007).

Various techniques which can be used to fabricate Al/ Si_3N_4 composite are stir casting, squeeze casting, spray casting, friction stir processing, hot forging, pressure infiltration, powder

metallurgy, etc. Among these processes, stir casting is a liquefied phase technique to fabricate metal matrix composites (MMCs) and is commercially used. It is simple, cheap, applicable to mass production with less production time, while it is superior to strengthen the materials by dispersing the considered reinforcement in the parent matrix.

Shoujiang et al. (2016) fabricated an Al alloy based composite through squeeze casting technique reinforced with 20 % Si_3N_4 by volume. Ramesh et al. (2012) prepared 6061/Al metal alloy based composites reinforced with Si_3N_4 particles by weight percentage coated with Ni through liquid metallurgy route. AMCs reinforced with Si_3N_4 (5-15%, by mass) particles developed by Sharma et al. (2016) through powder metallurgy technique. Sharma et al. (2015) manufactured Al/ Si_3N_4 composite via ball milling and solution mixing techniques. McEntire et al. (2016) reported that there is an increment in the hardness of the fabricated composites with varying Si_3N_4 ceramic nanoparticles by volume percentage. Rangari et al. (2013) depicted the influence of time on hardness of Al/ Si_3N_4 by aging heat treatment and found that 6061Al alloy is consuming considerably higher time to reach more hardness as compared to the fabricated composites. They showed maximum hardness with 15 wt. % of Si_3N_4 ceramic particles. Moreover, the published research works show that Al/ Si_3N_4 with different aluminium alloy matrix composites have been investigated by various researchers worldwide (Bocanegra-Bernal & Matovic, 2010). Though they prepared composites through different process but Al/ Si_3N_4 nanocomposites yet to be investigated through liquefied state.

Further, literatures show difficulty to manufacture with more contents of reinforcements in Al alloy composites through liquid route (stir casting) because of clustering of reinforced ceramic particles in the liquid state. Since, Si_3N_4 particles have poor wettability with liquid aluminium, Mg can be added to improve the wettability. Ball milling process and coating on the reinforcement also improve the wettability. Literature further show that adding of the Si_3N_4

ceramic particles with suitable wt. % develops various characteristics of the fabricated Al alloy based composites.

As a results of published literature, it has been found that there is lacunae in the fabrication and proper evaluation of Al/Si₃N₄ nanocomposites through liquefied state (stir casting). Therefore, the paper elaborates the production of Al/Si₃N₄ Nano-composites via liquefied state stir casting technique and the evaluation of physical, mechanical, and morphological characterization of fabricated nanocomposites are illustrated. Further, the novelty of the present work is an exertion to manufacture the material that can be employed in numerous parts of an automobile.

MATERIALS AND METHODS

The mechanical alloying and two-step stir casting methods were employed for fabricating Al/Si₃N₄ nanocomposites. Aluminium powder (APS 60-80 μm) and Si₃N₄ Nano-particles (40-65 nm of APS) were ball milled in the ratio 1:1 for milling time of 15 hours at 150 rpm with powder to ball ratio 1:10. Ball milling was accomplished in the inert (Ar) atmosphere in ball milled stainless steel cylinder in order to avoid the oxidation.

On the other hand, procured aluminium alloy ingot (A356) was melted in an electric resistance stir casting furnace. After melting, ball milled powders as discussed were dropped in crucible at slow rate. In the meantime, vortex is induced with the speed of 600±100 rpm for 8±2 minutes duration in two steps (Sambathkumar et al., 2017; Omrani et al, 2016). During the stirring, furnace was discharged in order to avoid any electric accident. After completion of stirring process, the liquid nanocomposites were poured into preheated (300-400°C) die at the pouring temperature of 680±30°C. The Nano reinforcement (Si₃N₄) was varied from 1-5% by weight in a step of 1%. The temperature of the molten metal was measured using thermocouple.

Specimens Preparation and Testing

Since the fabricated stir cast Al/Si₃N₄ nanocomposites are not the precise dimensions as required for testing. Therefore, the fabricated specimens were processed by machining operations (milling, grinding and/or turning) followed by emery papers of different sizes to make them suitable dimensions for the characterization. The specimen specifications with dimensions and tolerances were made as per ASTM standards (sheet type/sub-size) for different characterizations for metallic materials (Table 1). Moreover, for hardness and porosity, Samples of Al/Si₃N₄ nanocomposites were treated through machining operation followed by emery paper to make them suitable measurements. The theoretical and experimental densities were evaluated by rule of mixture (Equation 1) and Archimedes principle respectively.

$$\frac{1}{\rho_c} = \frac{1}{\rho_f} \left(\frac{w_f}{w_c} \right) + \frac{1}{\rho_m} \left(\frac{w_m}{w_c} \right) \quad (1)$$

where

ρ_c, ρ_m, ρ_f = densities of the composite, matrix and filler respectively;

w_c, w_m, w_f = weights of the composite, matrix and filler respectively.

The following method was adopted for porosity analysis.

$$\begin{aligned} \text{Porosity} &= \frac{\text{volume of pores}}{\text{volume of specimen}} = \frac{\text{weight of water absorbed}}{\text{density of water}} \\ &= \frac{\text{final weight of specimen after boiling in cc} - \text{initial weight of specimen in cc}}{\text{density of water in cc}} \\ &= \text{final wt. of sample after boiling in cc} - \text{initial wt. of sample in cc} \end{aligned} \quad (2)$$

Hardness analysis describes valuable data correlates to wear, mechanical and physical characteristics. Brinell hardness study was carried out for both Al as-cast and Al/Si₃N₄ nanocomposites. The ASTM E10-15a standard was used for hardness test. Figure 1 shows the detail steps of samples preparation and testing. Figure 2 shows the stir casting set up with different parts for casting composites sample whereas cast composites samples are shown in

figure 3. Five to seven composite samples for hardness test, three samples for tensile test, porosity test and density calculation were tested.

Table 1 Specification of Specimen

Name of Parts	Dimension of Specimen (mm)	
	ASTM E8M-13 (Flat)	Actual
Gage length (G)	50	50
Width (W)	12.5±0.25	10.75-13.0
Thickness (T)	0.13 ≤ T ≤ 19.05	4.0-5.5
Fillet radius (R)	6.25 min.	11.5-12.5
Overall length (L)	200 min.	195-200
Length of reduced section (A)	57 min.	59-60
Length of grip section (B)	50 min.	50-51
Width of grip section (C)	19.05 approx.	18.5-19.5

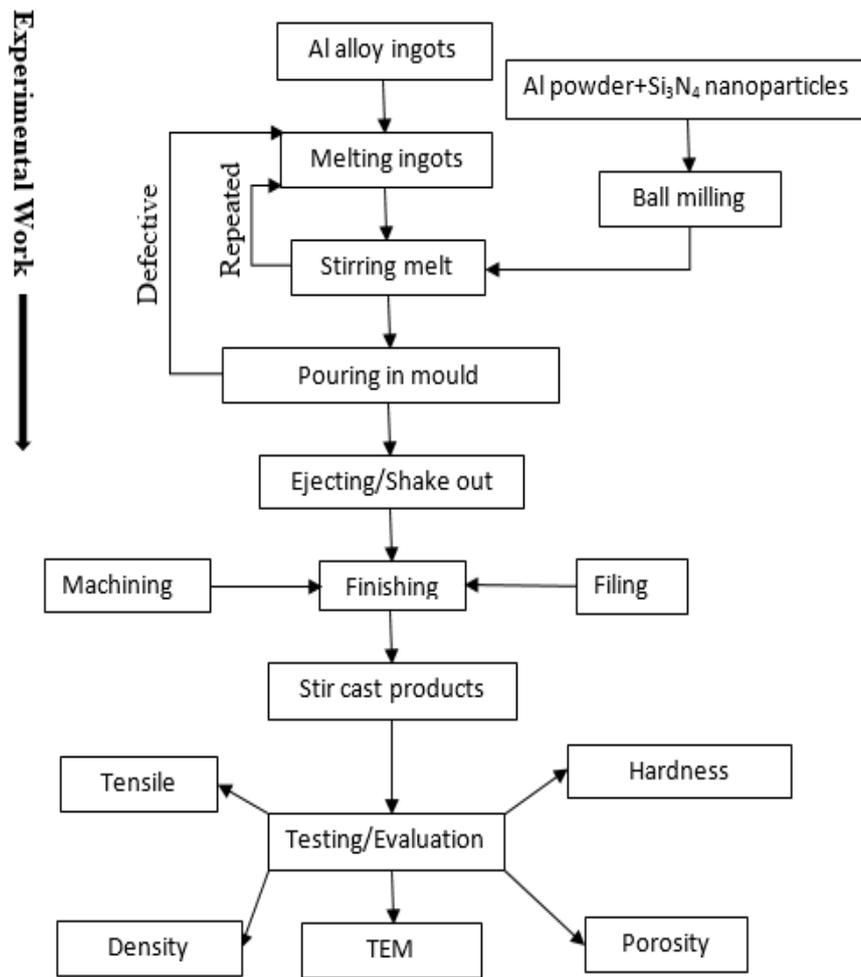


Figure 1 Steps for Preparation and testing of specimens

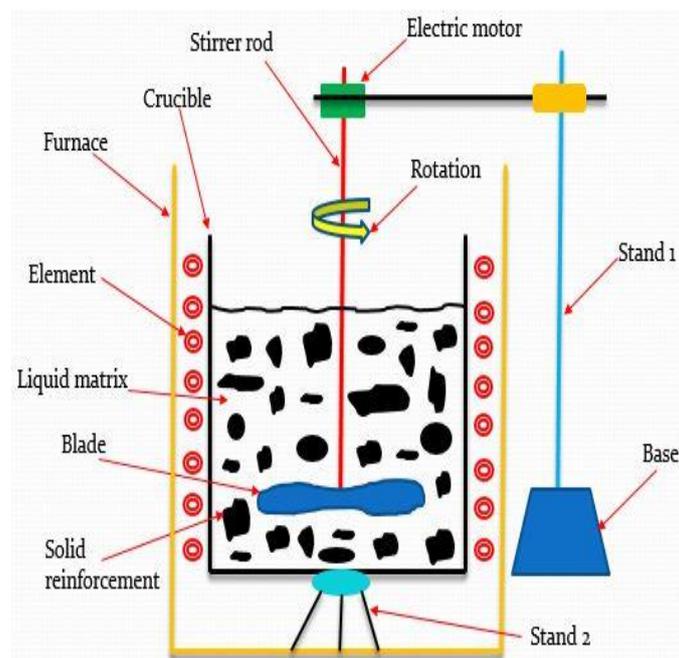


Figure 2 A block diagram of Stir Casting Set up

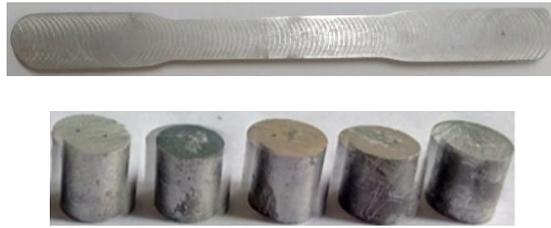


Figure 3 Stir cast Al/Si₃N₄ nanocomposites samples

RESULTS AND DISCUSSION

Physical Properties

Density and Porosity Evaluation

Figure 4 displays the effect of Si₃N₄ ceramic nanoparticles on density of the fabricated composites. From the graph, it can be observed that densities (experimental and theoretical) are changing on increasing the ceramic nanoparticles reinforcement. This is caused by the presence of Si₃N₄ ceramic particles which has density (3.17 g/cc) higher than A356 alloy (2.67 g/cc). An increase in the density of the composites has also been studied by the other researchers (Shalaby et al., 2017; Mazahery & Shabani, 2012) which also verify the present work. Further, the change in two densities are figured and found same trend. The increase in the densities can be attributed to the presence of the reinforcement nanoparticles in the composites.

The descriptions of influence of Si₃N₄ ceramic particles on porosity of Al/Si₃N₄ fabricated composites are revealed by the Figure 5. It is depicted from the graph that porosity changes by varying the Si₃N₄ ceramic reinforced particles. These pores are actually the open pores, i.e. pores found on the surfaces of the specimens and is known as apparent porosity. From the Figure 5, it is perceived that least porosity as 0.84% at 0 wt. % of Si₃N₄ while it is maximum as 1.12% at 5 wt. % of Si₃N₄ ceramic reinforced particles which shows less porosity as compared to previous once (Shalaby et al., 2017; Mazahery & Shabani, 2012; Tapaszto' &

Bala'zsi, 2010; Amigó, 2000). This expresses the better fabrication approach to produce the Al/Si₃N₄ composites through vortex technique via ball milling.

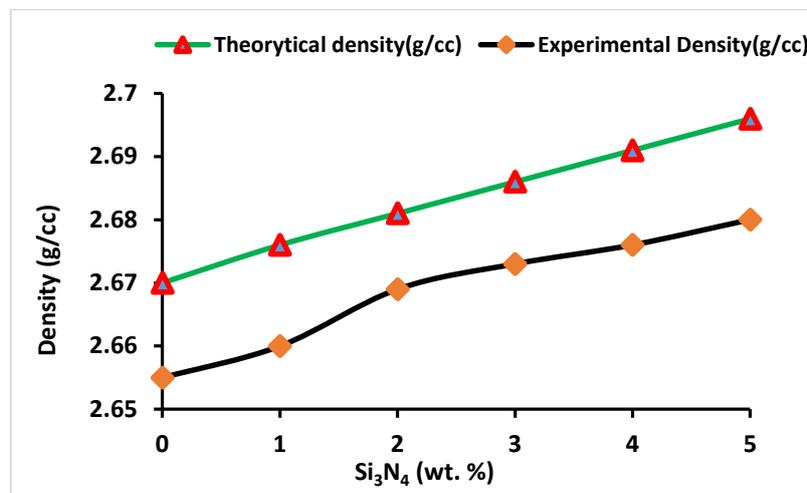


Figure 4 Density variation of Al/Si₃N₄ composites with wt. % of Si₃N₄

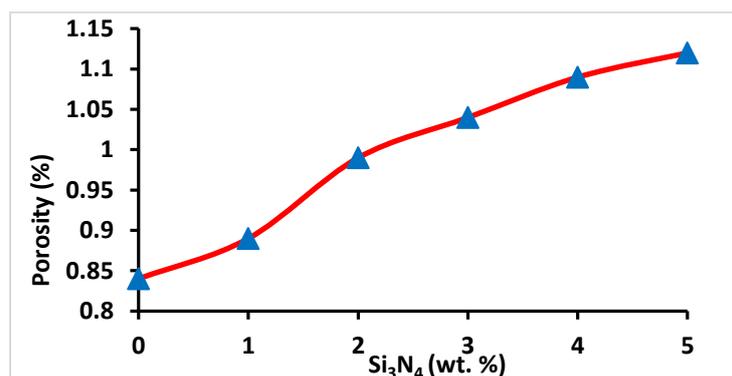


Figure 5 Porosity variation of Al/Si₃N₄ composites with wt. % of Si₃N₄

Mechanical Characterization

Hardness

Figure 6 illustrates influence of Si₃N₄ ceramic particles on hardness. The graph represents the increase in hardness on adding wt. % of Si₃N₄ ceramic reinforcement. Initially, its value is 43 and then increases to 86 HBN at 0 wt. % Si₃N₄ to 5 wt. % Si₃N₄ particles. In other words, hardness is increased by 100% with respect to the base alloy. The existence of Si₃N₄ particles which is brittle and hard as compared to A356 alloy is responsible for increasing the hardness of the said composites. Further, this also shows that there is enhancement of the wettability of the constituents in the composites by ball milling and vortex casting technique. Consequently,

improved mixing the constituents may have taken place in which reinforced particles get inserted in the composites (McEntire et al., 2016). Because of which dislocation movement reduces and increases mechanical properties. An improvement in hardness of Al6061-(n-Gr/Si₃N₄) and Al6082-(n-Gr/Si₃N₄) composites on adding Si₃N₄ nanoparticles have been predicted (Bocanegra-Bernal & Matovic, 2010). Presence of ceramics hard particles in Al alloy matrix significantly restricted to deform plastically consequences in the improvement of hardness of the composite. In the present study the maximum hardness of 86 HBN is depicted at 5 wt. % Si₃N₄ nanoparticles which is more than the hardness of Al/Si₃N₄ nanocomposites produced through stir casting reported (Sharma et al., 2015; Sharma et al., 2016).

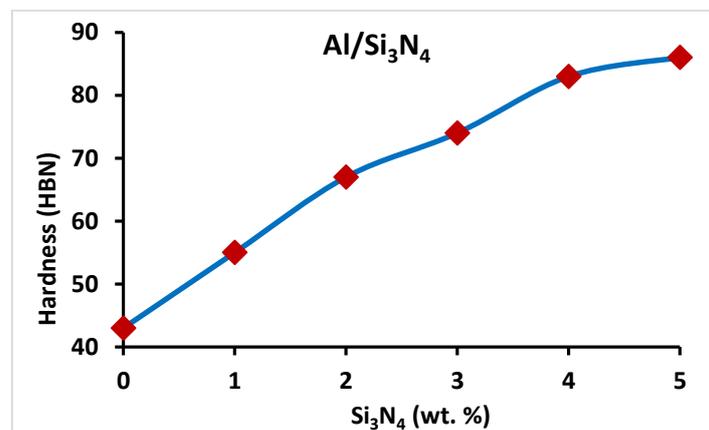


Figure 6 Hardness variation of Al/Si₃N₄ composites with Si₃N₄ particles

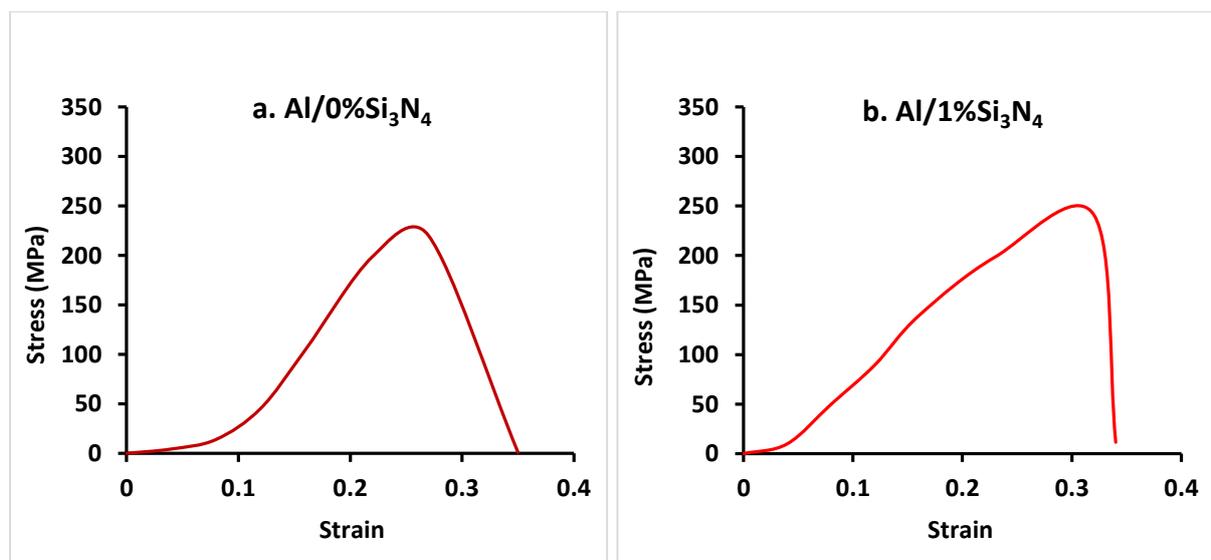
Tensile Strength

The tensile stress versus tensile strain for different wt. % of Si₃N₄ nanoparticles is shown in Figure 7(a-f). It is seen that tensile stress of composites increases on employing wt. % of Si₃N₄ nanoparticles. Further, it can be attributed that maximum stress is enhanced with the reinforcement of 5 wt. % of Si₃N₄ nanoparticles. Addition of hard and brittle ceramic reinforcements in the lightweight materials like Al and Mg based matrix enhances the overall performance of the composites. Moreover, it also shows that aluminium powder with Si₃N₄ nanoparticles are evenly dispersed in the whole molten matrix (base alloy). It can be deliberated that strength observed in the present study are comparable with the previous work.

Additionally, in the present case at 5 wt. % Si_3N_4 nanoparticles the maximum stress is depicted to 319 MPa which is more than the previous studies (Bhaskar et al., 2020; Radhika, 2018; Sharma et al., 2016; Sharma et al., 2015).

Morphological Characterization

The morphological analysis are revealed by transmission electron microscope (TEM) as shown in Figure 8(a-f). First of all, TEM image of Si_3N_4 nanoparticles is obtained as displayed in the Figure 8(a). To perceive the existence of ceramic Si_3N_4 particles in Al alloy matrix, composite samples (powder form) examined using TEM for 1-5% wt. of Si_3N_4 ceramic particles. Also, in order to identify the existence of Si_3N_4 ceramic particles, all TEM micrographs were analysed at the same magnifications. It can be observed from TEM images that there is the consistency of the ceramic Si_3N_4 particles. The average particle size (APS) of Si_3N_4 nanoparticles are found to be ranging 40-65 nm. The particle clustering has not found in the morphological study which also revealed by the previous results (Cai, 2020; Bhaskar et al., 2019). The even scattered reinforcement particles are observed even at 4% and 5% wt. of Si_3N_4 . Morphological analysis exhibits the occurrence of ceramic reinforced particles which are accountable for augmented mechanical properties.



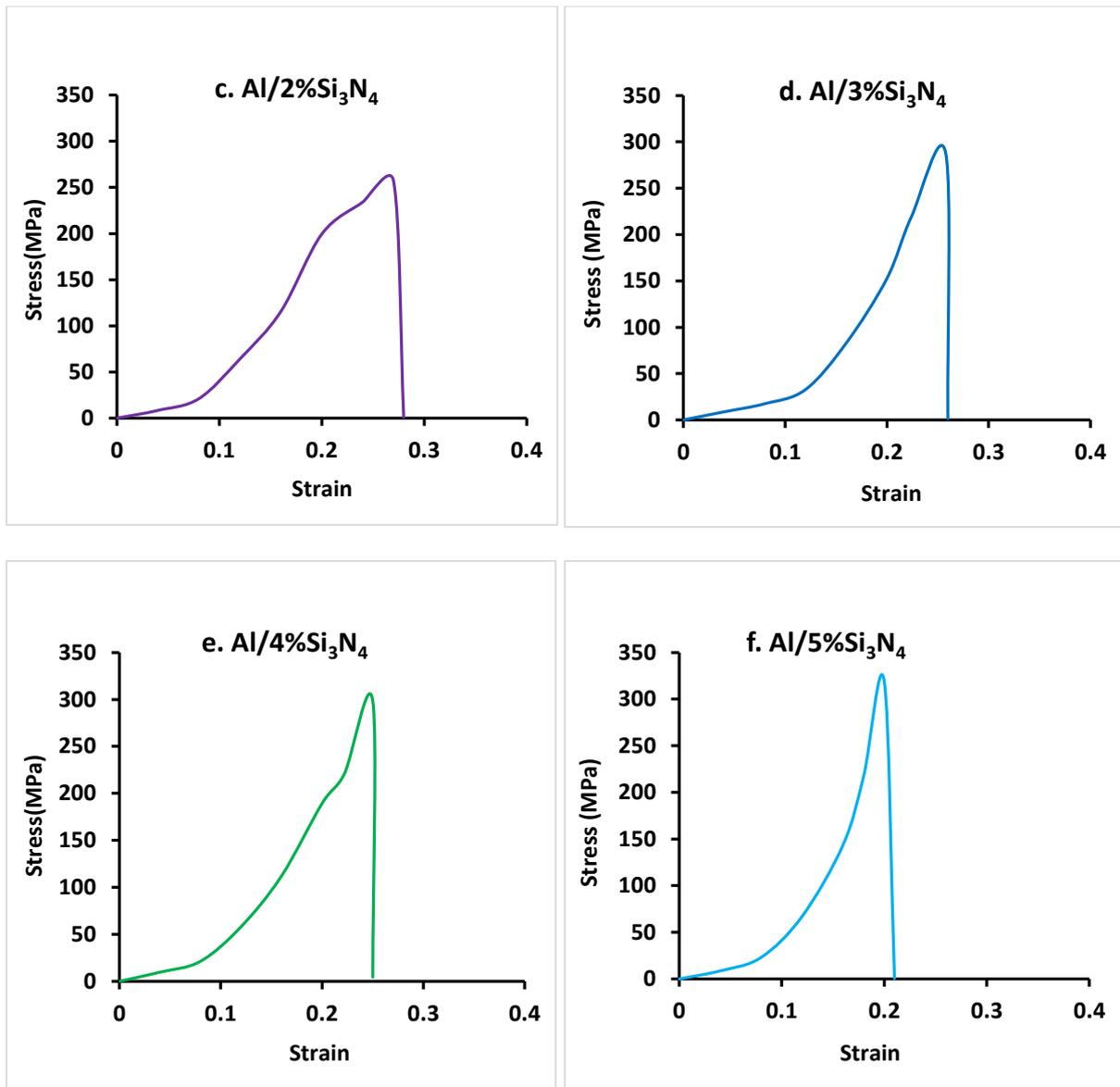


Figure 7(a-f) Tensile Stress Vs. Tensile Strain

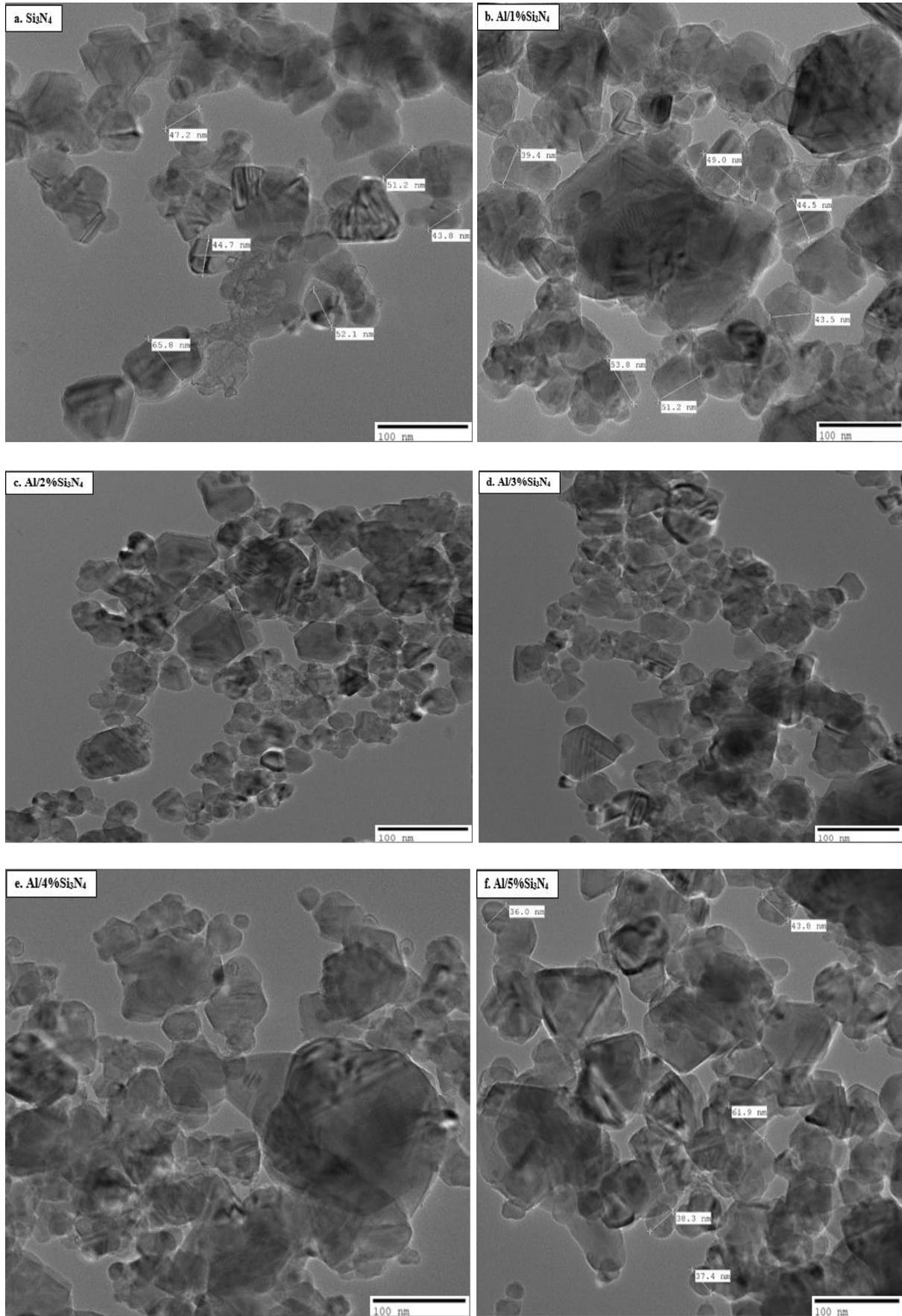


Figure 8(a-f) TEM images of Si_3N_4 and stir cast Al/ Si_3N_4 nanocomposites

Conclusions

A356/Si₃N₄ nanoparticles nanocomposites were made via liquefied state Stir Casting technique. The characteristics of the fabricated nanocomposites were evaluated and found to be considerably changed. The conclusions are summarized as below:

1. The incorporation of Si₃N₄ nanoparticles in the A356 alloy matrix exhibited a key feature in increasing mechanical and physical characteristics where Si₃N₄ nanoparticles were properly dispersed with A356 alloy matrix.
2. Physical characteristics such as density and porosity of nanocomposites are found to be linearly increased. The maximum porosity of 1.12% was predicted at 5 wt. % of Si₃N₄.
3. It is found that incorporation of Si₃N₄ nanoparticles enhances the mechanical properties of A356/Si₃N₄. Maximum tensile strength is depicted to be 319 MPa for A356/5%Si₃N₄ nanocomposites whereas hardness is increased from 43 HBN to 86 HBN.
4. The present morphological study revealed by TEM shows the presence of the Si₃N₄ nanoparticles in the fabricated nanocomposites. Morphology results did not show any appreciable particles clustering in the composites.

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References

- Amigó, V., Ortiz, J.L. & Salvador, M.D. 2000.** Microstructure and mechanical behavior of 6061Al reinforced with silicon nitride particles, processed by powder metallurgy. *Scripta Materilia*, **42**: 383–388.
- Bhaskar, S., Kumar, M. & Patnaik, A. 2020.** Effect of Si₃N₄ Ceramic Particulates on Mechanical, Thermal, Thermo-Mechanical and Sliding Wear Performance of AA2024 Alloy Composites. *Silicon*, **12**(11): 2020.
- Bhaskar, S., Kumar, M. & Patnaik, A. 2019.** Mechanical and Tribological overview of ceramic particulates reinforced aluminium alloy composites. *Reviews on Advanced Materials Science*. **58**(1): 280–294.

- Bocanegra-Bernal, M.H., & Matovic, B. 2010.** Mechanical properties of silicon nitride-based ceramics and its use in structural applications at high temperatures. *Materials Science and Engineering A*, **527**(6): 1314–1338.
- Cai, Q., Mendis, C.L., Chang, I.T.H., & Fan, Z. 2020.** Microstructure evolution and mechanical properties of new die-cast Al-Si-Mg-Mn alloys. *Materials and Design*, **187**: 108394.
- Dai, Z., Zhu, S., Liu, S., & Guan, S. 2012.** Preparation and properties of high-performance polyethersulfone composites reinforced by modified silicon nitride. *High Performance Polymer*, **24**: 609–615.
- Kumar, R.A. & Devaraju, A. 2021.** Modeling of Mechanical Properties and High Temperature Wear Behavior of Al7075/SiC/CRS Composite Using RSM. *Silicon*, **13**: 3499–3519.
- Kumar, N.M., Kumaran, S.S. & Kumaraswamidhas, L.A. 2016.** Wear behaviour of Al 2618 alloy reinforced with Si₃N₄, AlN and ZrB₂ in situ composites at elevated temperatures. *Alexandria Engineering Journal*, **55**(1): 19-36.
- Mazahery, A. & Shabani, M.O. 2012.** Nano-sized silicon carbide reinforced commercial casting aluminium alloy matrix: Experimental and novel modeling evaluation. *Powder Technology*, **217**: 558-565.
- McEntire, B.J., Lakshminarayanan, R., Thirugnanasambandam, P., Sampson, J.S., Bock, R., & Brien, D.O. 2016.** Processing and Characterization of Silicon Nitride Bioceramics. *Bioceramics Development Applications*, **6**(1): 1–9.
- Omrani, E., Moghadam, A.D., Menezes, P.L. & Rohatgi, P.K. 2016.** Influences of graphite reinforcement on the tribological properties of self-lubricating aluminum matrix composites for green tribology, sustainability, and energy efficiency—a review. *International Journal of Advance Manufacturing Technology*, **83**: 325–346.
- Parveen, A., Chauhan, N.R. & Suhaib, M. 2019.** Study of Si₃N₄ reinforcement on the morphological and tribo-mechanical behaviour of aluminium matrix composite. *Materials Research Express*, **6**(4): 042001.
- Qu, S., Feng, A., Geng, L., Shen, J. & Chen, D. 2020.** Silicon Nitride Whisker-Reinforced Aluminum Matrix Composites: Twinning and Precipitation Behavior. *Metals* **10**: 420.
- Radhika, N. 2018.** Comparison of the mechanical and wear behaviour of aluminium alloy with homogeneous and functionally graded silicon nitride composites. *Science and Engineering of Composite Materials*, **25**(2): 261-271.
- Ramesh, C.S., Keshavamurthy R., Channabasappa, B.H., & Pramod. S. 2010.** Friction and wear behavior of Ni-P coated Si₃N₄ reinforced Al6061 composites. *Tribology International*, **43**: 623-634.
- Sambathkumar, M., Navaneethakrishnan, P., Ponappa, K. & Sasikumar, K.S.K. 2017.** Mechanical and Corrosion Behavior of Al7075 (Hybrid) Metal Matrix Composites by Two Step Stir Casting Process. *Latin American Journal of Solids and Structures*. **14**: 243-255.

- Selvam, J.D.R., Dinaharan, I., Vibin Philip, S. & Mashinini, P.M. 2018.** Microstructure and mechanical characterization of in situ synthesized AA6061/(TiB₂+Al₂O₃) hybrid aluminum matrix composites. *Journal of Alloys and Compounds*, **740**: 529-535.
- Shalaby, E.A.M., Churyumov, A.Y., Besisa, D.H.A., Daoud, A. & Abou El-Khair, M.T. 2017.** A Comparative Study of Thermal Conductivity and Tribological Behavior of Squeeze Cast A359/AlN and A359/SiC Composites. *Journal of Materials Engineering and Performance*, **26**(7): 3079—3089.
- Sharma, N., Khanna, R., Singh, G. & Kumar, V. 2016.** Fabrication of 6061 aluminum alloy reinforced with Si₃N₄ / n-Gr and its wear performance optimization using integrated RSM-GA, *Particulate Science and Technology*, **35**(6): 731-741.
- Sharma, P., Sharma, S. & Khanduja, D. 2016.** Production and Characterization of AA6082- (Si₃N₄ + Gr) Stir Cast Hybrid Composites. *Particulate Science and Technology*, **35**(2): 158-165.
- Sharma, P., Sharma, S., & Khanduja, D. 2015.** Production and some properties of Si₃N₄ reinforced aluminium alloy composites. *Journal of Asian Ceramic Society*, **3**: 352–359.
- Sharma, P., Sharma, S. & Khanduja, D. 2015.** Production and some properties of Si₃N₄ reinforced aluminium alloy Composites. *Journal of Asian Ceramic Societies*, **3**: 352–359.
- Shettar, M., Hiremath, M.P., Shankar, G., Kini, A. & Sharma, S. 2021.** Tribolayer Behaviour and Wear of Artificially Aged Al6061 Hybrid Composites. *International Journal of Automotive and Mechanical Engineering*, **18**(2): 8668 – 8676.
- Suresh, S., Gowd, G.H. & Deva Kumar, M.L.S. 2019.** Mechanical Properties of AA 7075/Al₂O₃/SiC Nano-metal Matrix Composites by Stir-Casting Method. *Journal of The Institution of Engineers (India): Series D*, 43–53.
- Tapasztó, O. & Bala'zsi, C. 2010.** The effect of milling time on the sintering kinetics of Si₃N₄ based Nanocomposites. *Ceramics International*, **36**: 2247–2251.
- Vijaya, K., Rangari, M., Yousuf, S., Jeelani, 2013.** Influence of SiC/Si₃N₄ Hybrid Nanoparticles on Polymer Tensile Properties. *Journal of Composites*, **2013**: 1-11.
- Wang, S.R., Wang, Y.Z., Wang, Y., Geng, H.R. & Chi, Q.S. 2007.** Microstructure and infiltration kinetics of Si₃N₄/Al-Mg composites fabricated by pressureless infiltration. *Journal of Materials Science*, **42**: 7812-7818.
- Zheng, K.L., Wei, X.S., Yan, B., & Yan, P.F. 2020.** Ceramic waste SiC particle-reinforced Al matrix composite brake materials with a high friction coefficient. *Wear*, **458-459**: 203424.