Nano World Journal

https://doi.org/10.17756/nwj.2023-s3-123

Research Article

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Characterization and Dispersion Analysis of Silicon Carbide Nanoparticles in Polypropylene for Metal Matrix Composites

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Received: July 28, 2023
Accepted: October 27, 2023
Published: October 31, 2023

Citation: Sapaev IB, Jasim LH, Mahalakshmi, Subramani V. 2023. Characterization and Dispersion Analysis of Silicon Carbide Nanoparticles in Polypropylene for Metal Matrix Composites. *Nano World J* 9(S3): S685-S691.

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Abstract

The possibility of using polypropylene (PP) to embed silicon carbide nanoparticles (SiCnp) into metal matrix composites (MMCs) was explored. The modified Hummers' method was used to manufacture the SiCnp, and X-ray photoelectron spectroscopy (XPS) and X-ray diffraction (XRD) were used for characterization. The nanocomposites of PP and SiCnp were created by means of the solution mixing method. Nanoindentation was used to map the elastic modulus and tension and nanoindentation to quantify and qualify the dispersion of SiCnp in PP. Excellent mechanical properties were produced by the results, which showed that the SiCnp were well dispersed in the PP matrix. The quantification study verified a good dispersion degree due to the meticulous management of the energy components and dispersion time. In addition, early research confirmed that PP was an efficient inclusion vehicle for metallic matrices containing nano reinforcements.

Keywords

Silicon carbide nanoparticles, Dispersion degree, Polymer matrix composites, Sandwich technique, Materials processing

Introduction

Carbonaceous nano reinforced MMCs are very desirable in many fields, but notably in aerospace and aviation. Composites with low density and high strength, adaptability to various manufacturing procedures, and the potential to achieve significantly better mechanical behaviour with nano reinforcements are just a few of the appealing properties of these materials [1, 2]. Powder metallurgy, liquid state techniques, electrochemical deposition, sputtering deposition, molecular mixing, mechanical milling, and many more standard methods have been employed to create nano reinforced MMCs [3, 4]. But there are a few problems with these processes that crop up throughout production, and those problems could affect the mechanical properties and characteristics of the materials. Inadequate dispersion of nano reinforcements, reinforcement damage, cluster formation, and matrix-fiber interactions are among these challenges [5, 6].

Processing nano reinforced aluminum matrix composites (NRAMCs) requires careful attention to the reinforcement dispersion since clustering and porosity might result from an excess of nano reinforcements in the nanocomposite. To improve the nanocomposites' dispersion degree, some writers have employed powder metallurgical procedures. As an example, AA5083 aluminum powders combined with multiwalled carbon nanotubes (MWCNTs) were investigated [7]. After a mechanical dispersion process, the composites were sintered and extruded

to bring the MWCNTs into alignment with the metallic matrix and to remove any porosity. Both the maximum stress and yield strength were found to be greatly raised when 1.5 wt.% MWCNTs were added during high-energy ball milling.

The composites sintered at 550 °C were created by authors [7] by employing an *in-situ* growth technique of carbon nanotubes (CNTs) in aluminum powders. They found that 0.8 wt.% CNTs was the best point for improving mechanical parameters including hardness and strength; further increases in CNT content led to decreases in mechanical capabilities. In order to create aluminum-CNT composites, authors [8] used ball milling to mix and distribute tungsten and alumina balls throughout the aluminum matrix. A microwave sintering method was used to cool compact the milled powders before they were sintered. The scientists observed that using alumina balls for milling the samples increased the dispersion degree of CNTs into the metal matrix, leading to a somewhat larger hardness improvement than using tungsten balls. Synthesizing magnesium composites with the hybrid aluminum-CNT reinforcement allowed to enhance the nano reinforcements' dispersion. Traditional methods of solid-state powder metallurgy were employed in a two-stage process. A composite of aluminum powder and CNTs was first combined, and then magnesium powder was added to the mixture. The near-dense magnesium nanocomposites with a combination of nano-powdered aluminum-CNTs were produced by combining microwave-assisted fast sintering with hot extrusion. A higher percentage of aluminum in the hybrid composite resulted in improved mechanical properties such ultimate strength, yield strength, and elastic modulus [9].

MMCs have been synthesized by other authors using the melting and solidification technique. As an example, composites based on magnesium were manufactured by authors [10] using the disintegrating melt deposition approach. By combining AZ31 and A5083 alloys reinforced with CNTs via disintegrating melt deposition and extrusion, authors were able to enhance the mechanical characteristics of the composite. However, all of the mechanical properties remained unchanged, with the exception of ductility, which showed an increase as the percentage of CNTs increased.

A technique developed involves functionalizing CNTs with PP, which in turn generates van der Waals forces. These forces facilitate a solid interface between the nano reinforcements and the aluminum powder matrix. The CNTs are evenly distributed throughout the matrix using this method, which also involves pyrolyzing the polymer and pressing and sintering the residual material. Some authors have included nano reinforcements into metallic matrices using this polymer as a vehicle or inclusion medium. For this inclusion vehicle to scale from the lab to the factory, it needs to be easy to disperse, have a low fusion temperature, and be inexpensive to process; these factors will ultimately show up in the NRAMCs mechanical properties. Nano reinforced PMCs with SiCnp have been the subject of numerous investigations, with a variety of dispersion methods employed. PP has demonstrated positive dispersion results and significant improvements in the mechanical responses of its composites [11, 12]. It is a hydrophilic, biocompatible, and nontoxic polymer with excellent film-forming capabilities. Molecular dispersion of nano reinforcements greatly improves mechanical characteristics, thermal stability, and electrical conductivity [4, 13]. In order to make MMCs using the sandwich approach, sheets of the metal to be reinforced are stacked with PP reinforced layers, and then the whole stack is pyrolyzed [14]. In this study, we will apply this method because it consistently yields high-quality composite materials.

Having an appropriate distribution of reinforcement is crucial, as all researchers agree. One model that has been created for quantitatively assessing dispersion degree is based on feature size, where the relative particle size ratio indicates clustering. An approach to feature local concentrations analysis known as the quadrat technique uses characteristic-sized small quadrats (elements) to establish the dispersion with standard deviation/variance of feature concentrations. Due to its simplicity and generally accurate judgement on dispersion when applied correctly, variants of the quadrat approach find utility in numerous fields. This method measured the extent to which CNTs were dispersed within the matrix. The methodology proved to be an effective nano reinforcement dispersion index, and their results were comparable to those of the ASTM standard index for matrix inclusion dispersion. The distances between neighboring nano reinforcements follow a Poisson distribution when they are dispersed randomly on a surface. Lastly, the method presupposes that, provided that all inclusion particles are dispersed at an equal free-path distance, the dispersion will be ideal.

In order to attain desirable qualities for PMCs nano reinforced with SiCnp, two important parameters influencing the mechanical behaviour of NRAMCs must be taken into account. The first step is to ensure high-quality SiCnp, which are influenced by the synthesis technique and the conditions for exfoliation and reduction [15]. Additionally, the mechanical reaction of the PMCs is impacted by the SiCnp's dispersion into the matrix. According to some research, the enhanced characteristics of the resulting nanocomposites are hindered by the cluster formation of SiCnp. Using PP as a nano reinforcement inclusion vehicle, produced MMCs with a high degree of dispersion in both the polymeric and metallic matrices. In order to approximate the dispersion of nano reinforcements into a matrix, statistical and numerical analysis were employed with the D0.1 distribution model. Incorporating SiCnp into a metallic matrix was achieved in the present study by means of PP. The goal is to study SiCnp dispersion in the PP matrix, which is thoroughly assessed by measuring and qualifying dispersion. An improvement in mechanical characteristics would indicate that the dispersion procedure was successful.

This research is anticipated to aid in the creation of scalable manufacturing procedures for NRAMC fabrication that are both economical and efficient. The solution mixing approach will be used to make the PP/SiCnp composites [16].

Materials and Methods

Synthesis of SiCnp

The modified Hummers' method was used to synthesis graphite oxide (GrO) from commercial graphite powder with

a size less than 20 µm (Figure 1). A combination of graphite and $NaNO_3$ was modified by adding concentrated H_2SO_4. By employing a water bath and a water-cooled condenser, the reaction temperature was maintained below 20 °C while the mixture was cooled down. Afterwards, $KMnO_4$ was gradually added. The addition of 3% H_2O_2 gradually followed three hours of stirring the liquid, producing a strong exothermic reaction. After stirring for 30 min, the mixture was centrifuged at 4000 rpm for 15 min, and the liquid on top was removed by removing the cap. After centrifugation, the residual GrO was rinsed with distilled water. We kept doing this until we reached a pH of neutral. The GrO sludge was ground into a powder that was less than 40 µm in size after being dried in a rotary evaporator and subjected to ball milling. An electrical furnace was used to produce the SiCnp by thermally exfoliating and reducing GrO in a quartz glass reactor fashioned like a tube. An initial degassing under N_2 flow (100 ml/min) at room temperature for 30 min was part of a one-step heating procedure. Following that, the sample was washed at 400 °C with a flow rate of 100 ml/min of N_2. The last step was to let the furnace cool to ambient temperature.

Synthesis of PP/SiCnp nanocomposites

The PP/SiCnp nanocomposites were synthesized using the same settings as in earlier studies [17]: 4 wt.% PP was achieved by diluting hydrolyzed PP pellets in hot distilled water. Following this, the SiCnp were added to the PP solution at concentrations of 0.3 wt.% and 0.60 wt.%, respectively. Using magnetic stirring at 600 - 800 rpm, the SiCnp were mixed into the PP solution until they were evenly distributed. We next used a high-frequency ultrasonic lance set to 40% of its maximum power for three hours to vigorously agitate the PP/SiCnp mixture. A 15 cm long and 3 cm wide acrylic mold was filled with the PP/SiCnp solution, and then left to cure at room temperature for seven days (Figure 2).

Manufacturing of the NRAMCs

A combination of commercially available AA1100 sheets (99% pure, 0.3 mm thick) and nanocomposites of PP and SiCnp (0.14% thick) were employed in the sandwich approach to create the NRAMCs. The aluminum sheets were arranged in a stack with two PP/SiCnp composite sheets on top of each other. The aluminum sheets were able to connect through diffusion because the PP was thermally damaged during the heated compression of the stacking in a die in an argon atmosphere, which prevented the SiCnp from oxidizing. Beginning at ambient temperature, the temperature was raised to 645 °C and the pressure to 30 MPa in a step-by-step fashion. The next step was to wait 1.5 h for the PP to evaporate and the aluminum to disperse. The selection of these values was informed by the thermogravimetric investigation of the PP/SiCnp nanocomposites.

Using a Mettler Toledo TGA/SDTA 851, a thermogravimetric balance, we were able to determine the thermal deterioration of the PP/SiCnp. The sample, which weighed around 10 mg, was placed in a ceramic crucible and heated under an N_2 environment at a rate of 10 °C/min up to 1000 °C (50 ml/min). The schematic approach for the fabrication of NRAMC is shown in figure 3.

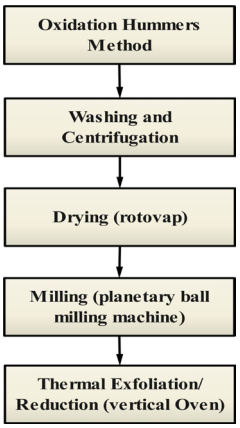


Figure 1: Schematic procedure of the SiCnp synthesis.

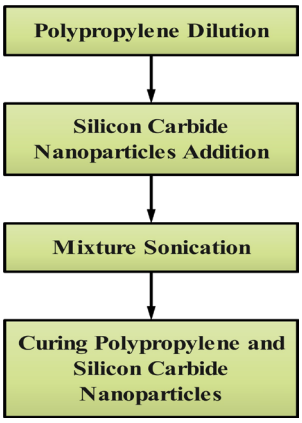


Figure 2: Synthesis of PP/SiCnp nanocomposites.

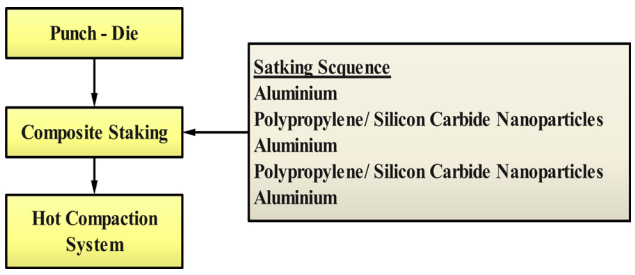


Figure 3: Synthesis of the NRAMCs.

Characterization of SiCnp

XPS on Specs benchmark equipment with a PHOIBOS 150 1D-DLD analyzer was employed for the surface chemical characterization of the SiCnp.

Dispersion quantification of nano reinforced PMCs

For the nanoindentation process, a Berkovich diamond tip was utilized with an IBIS Authority Fischer-Cripps nanoindenter. The elastic modulus was evaluated in a 2.5 x 2.5 μm^2 delimited region; three distinct areas were checked for each sample, using a maximum force of 0.8 mN and an indentation depth of around 50 - 200 nm. The $D_{0.1}$ distribution model was used for the analysis of these pictures. In addition to the horizontal and vertical axes, they were also divided into 10 x 10 grid lines. Next, the distance between the closest elastic mod-

ulus variations at each grid intersection were measured, both horizontally and vertically. The outcomes of the data analysis statistical distribution model are shown in equation 1.

$$f(x) = \frac{1}{xn\sqrt{2\pi}} \exp\left[\frac{-1}{1}\left(\frac{\ln x - m}{n}\right)^2\right] for\ x > 0 \quad (1)$$

Where $m = \ln\frac{\mu^2}{\sqrt{\mu^2+\sigma^2}}$ and $n = \sqrt{\ln\frac{\mu^2+\sigma^2}{\mu^2}}$, x denotes the distance along the free path, μ is the average of the dispersion, and σ is the standard deviation of the measured free-path distance.

A dispersion parameter, $D_{0.1}$, was utilized for dispersion quantification. The probability of the free-path distance distribution being within μ (0.9 - 1.1 μ) of the mean is the definition of this parameter. Usually, the distribution of distances follows a lognormal distribution model, as shown in equation 2 for $D_{0.1}$.

$$D_{0.1} = 1.1539 \times 10^{-2} + 7.5933 \times 10^{-2} \times \left(\frac{\mu}{\sigma}\right) + 6.6838 \times 10^{-4} \times \left(\frac{\mu}{\sigma}\right)^2 - 1.9169 \times 10^{-4} \times \left(\frac{\mu}{\sigma}\right)^3 + 3.9201 \times 10^{-6} \times \left(\frac{\mu}{\sigma}\right)^4 \quad (2)$$

If the value of the parameter, $D_{0.1}$ is 100%, then all the reinforcements are equally spaced, regardless of their distance. In other words, the values of the parameter can be the same for varying percentages of nano reinforcements added. More data about spacing is near the mean μ when the value is higher, D-0.1.

Mechanical properties of nano reinforced PMCs

Nanoindentation tests were also carried out using a peak load of 5 mN. At this load, the bulk properties of PP/SiCnp composites can be obtained. Tensile tests were also performed on the thin sheets of the PP/SiCnp composites under the ASTM D882 standard; an INSTRON 5582 universal testing machine at a rate of 10 mm/min was used.

Results and Discussion

Pure graphite, GrO, and SiCnp are depicted in figure 4 by means of their respective XRD patterns. The d002 reflection of graphitic carbon is a strong and compact peak at 26°. The graphite's oxidation causes a broadening of the peak and a shift to lower 2θ angles for the SiCnp. One possible explanation for these results is that graphite's crystallinity is decreasing as a result of the steady addition of oxygen to its structure. The specific surface area and the existence of residual oxygen groups on the surface are two important parameters

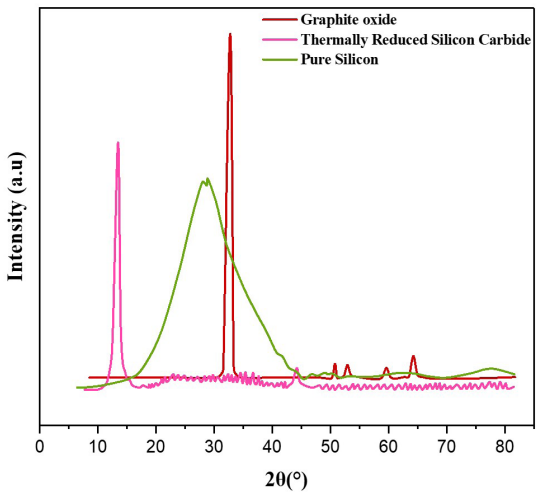


Figure 4: XRD patterns of graphite, GrO, and SiCnp.

that greatly impact the dispersion state of silicon. Improving the dispersion of nano reinforcements in polymeric matrices is greatly influenced by these parameters [9].

At approximately 10°, there is a surprisingly broad peak that represents the extended GrO. The GrO might have been SiCnp, as the d002 peak moved to greater 2θ angles and reached a value of approximately 22.5°.

Table 1 shows the deconvoluted XPS data. By examining the C1s and O1s photoelectric lines, we were able to identify the chemical compositions and measure the carbon and oxygen atomic percentages on the nano reinforcements' surface. The efficacy of the oxidation process during the Hummers' method is demonstrated by the fact that SiCnp are constituted of 84.6% carbon and 15.4% oxygen. C-O bonds, which can represent ether, alcohol, or epoxy functional groups, are the most important part of SiCnp nano reinforcements.

Strong hydrogen-bonding interactions between the oxygen-containing functional groups of SiCnp and the hydroxyl groups of PP make SiCnp sheets completely compatible with PP chains, in addition to improving the barrier properties, mechanical interlocking, and load transfer within the matrix [18]. Consequently, as we will see in the following sections, the nanocomposites' mechanical characteristics ought to be enhanced. However, due to the thermal reduction process, the contribution of various oxygen groups reduces. Nano reinforcements with functional groups on their surfaces have great promise as light metallic material reinforcements because of

Table 1: Deconvoluted XPS data for C1s and O1s.

Peak C1s (eV)	Area (%)	Bonding environment	Peak O1s (eV)	Area (%)	Bonding environment	OXPS (wt.%)	C/O
284.5	63.0	Silicon	530.2	9.0	Quinones	15.4	5.5
285.3	14.8	C-C/C-H sp^3	531.3	20.9	C-O carbonyl/carboxyl		
286.2	13.7	C-O	532.3	20.8	Hydroxyl/epoxy		
287.4	4.4	C-O	533.5	42.6	C-O, C-OH, C-O-C phenol/ether		
288.6	3.3	O-C-O	534.7	6.7	H_2O physisorbed/chemisorbed		
289.9	0.8	sp^2 C shakeup					

their favorable interactions with metallic matrices. Because of the strong hydrogen-bonding connection between aluminum and GrO made possible by the hydroxyl groups in PP, SiC nanosheets can be more effectively adsorbed onto aluminum by using PP [19].

Dispersion of SiCnp in PP

As mentioned earlier, the fabricated PMCs had an elastic modulus of up to 0.8 mN. The property's value is lower in darker colors, which means the measurement was taken in a non-nano reinforced area. The presence of nano reinforcement, or at least some of it, is shown by lighter spots (due to the size of the SiCnp, some parts of the nano reinforcements can be close to the surface and others beneath the surface). The greater light spots in the mapping contour of the composite with 0.6 wt.% SiCnp are not surprising. Both pictures indicate a uniform distribution of the characteristic, which is an indirect indication of how evenly distributed the nano reinforcement is throughout the polymeric matrix.

In the composite reinforced with 0.3 wt.% SiCnp, the dispersion model's frequency distribution reveals an average distance of 0.423 μm and a dispersion degree $D_{0.1}$ of 11.25%; the majority of the data was gathered around 0.2 μm. As shown in figure 5 for the composite that was reinforced with 0.6 wt.% SiCnp (Figure 6), the average distance was 0.270 μm and the dispersion degree $D_{0.1}$ was 8.09%. The most extensive data collection was done near 0.090 μm. We expected these results because their presence is increased with increasing reinforcement quantities in the matrix. The consistency of the dispersion degree percentages was confirmed by earlier research that revealed similar amounts of dispersion. A method for estimating the degree of particle dispersion in a matrix was investigated using free-path distance [3]. Possible discrepancies in the sonication process explain why their dispersion degree of approximately 3 - 4% is lower than the results observed in this investigation. The bulk of the cited studies fail to provide any quantitative measures for the nano reinforcement's distribution throughout the polymeric matrix. The solution mixing technique to create composite films by adding varying amounts of silicon oxide (2 wt.% and 4 wt.%) to PP. No quantitative analysis of nano reinforcement dispersion was provided by the authors, despite their evaluation of the mechanical properties, especially the plasticity, of the composites. In similar, used methanol to disperse their poly(vinyl acetate) solution before combining it with another solution. There was no specified mechanism for quantifying the dispersion of the nano reinforcements in the polymeric matrix, although they did note that the nanocomposite was more flexible, indicating better dispersion.

Mechanical characterization of the PP/SiCnp nanocomposites

In this case, a maximum load of 5 mN was applied during conventional quasi-static nanoindentation testing to demonstrate the impact of nano reinforcement on the mechanical properties of PP/SiCnp. Additionally, this test provided a dispersion qualification of the nano reinforcements into the PP matrix, meaning that if the composites had a satisfactory

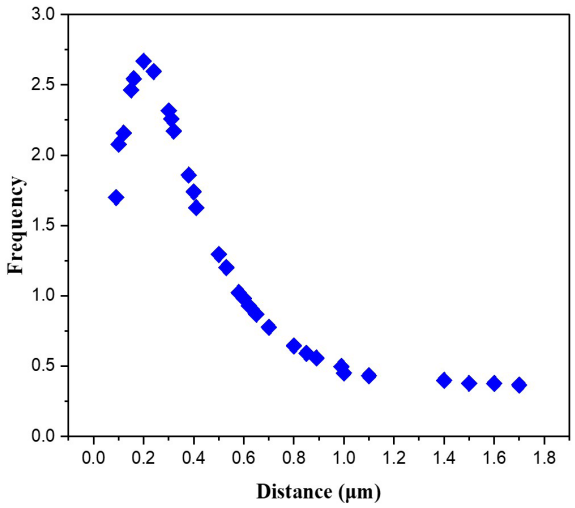


Figure 5: Frequency distribution for the PP/SiCnp composite with 0.3 wt.%.

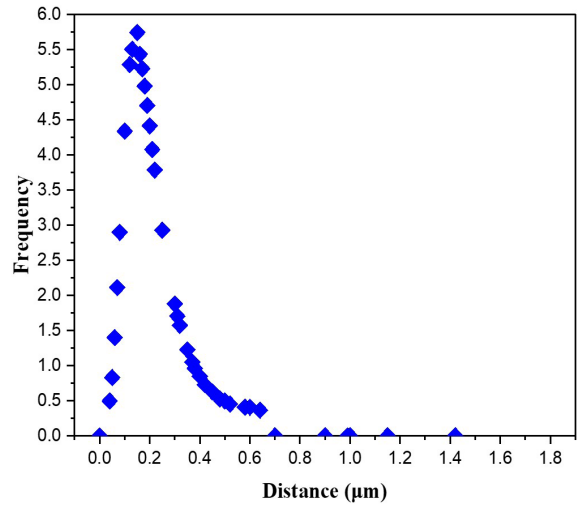


Figure 6: Frequency distribution of the PP/SiCnp 0.6 wt.% composite.

mechanical reaction, it would be because the nano reinforcements were well-dispersed into the matrix. It is expected that the penetration depth will decrease with increasing SiCnp concentration due to the tougher reinforcing phase, as seen in figure 7a. The addition of reinforcement increases the elastic modulus by an additional 162% for 0.6 wt.% SiCnp, as shown in figure 7b, as compared to the unreinforced material (0 wt.% SiCnp). In contrast, the composite reinforced with 0.3 wt.% SiCnp exhibits an increase in hardness before beginning to exhibit a decline, as displayed in figure 7c. Indirectly, the results imply that the dispersion conditions are sufficient up to 0.3 wt.% SiCnp, but that for larger levels, proper dispersion is not possible. The researchers found that the mechanical properties of the material decreased due to factors like the dispersion of the nano reinforcement and its affinity with the polymeric matrix.

The yield strength and ultimate tensile strength that were measured during tensile tests are displayed in figure 8. The yield strength value increased by 49% when 0.3 wt.% SiCnp were added, but it reduced when 0.6 wt.% SiCnp were added (Figure 8a). For the ultimate tensile strength (Figure 8b), a

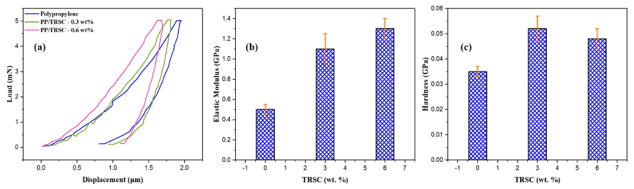


Figure 7: The outcomes of the nanoindentation experiments conducted on the composites made of PP and SiCnp: **(a)** load-displacement curve, **(b)** elastic modulus, and **(c)** hardness.

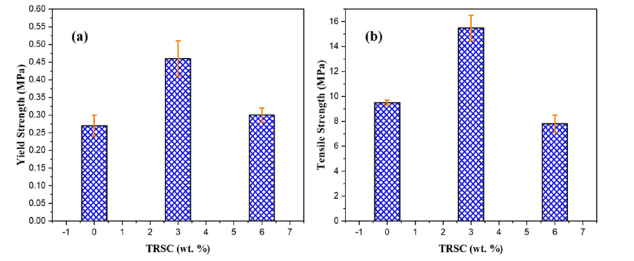


Figure 8: Tensile mechanical properties of PP/SiCnp composites: **(a)** yield strength and **(b)** ultimate strength.

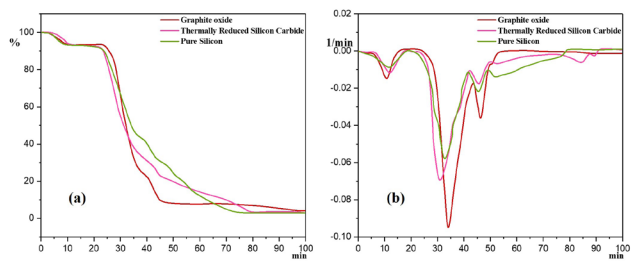


Figure 9: TGA-DTG analysis for PP/SiCnp nanocomposites.

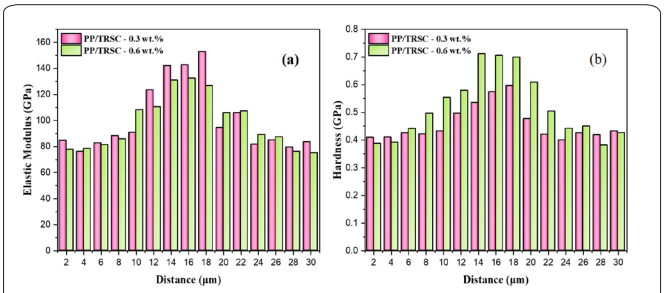


Figure 10: (a) Elastic modulus and **(b)** hardness by nanoindentation tests.

concentration of 0.3 wt.% SiCnp resulted in a 54% increase, whereas the addition of 0.6 wt.% SiCnp caused a substantial drop. After a certain content, the properties start to degrade for reasons unrelated to the reinforcement dispersion [7]. These include things like pore entrapment during polymerization, which is caused by the exothermic reaction of the polymer the sample gets hot during sonication, and the water evaporates, which impacts the mechanical response of the composites. Note that the magnitude of the interaction volume impacts the outcomes of both the nanoindentation and tensile tests when comparing their respective results. The results obtained in nanoindentation tests do not immediately reflect on the tensile test since, when the tip of the test is put on a pore, the reading is considered unusual and rejected.

Preliminary study for NRAMCs

It is necessary to know the thermal deterioration of the PP/SiCnp composites before making the NRAMCs utilizing the sandwich technique. In figure 9, the thermogravimetric analysis (TGA-DTG) findings are displayed for the polymeric composites reinforced with 0, 0.3, and 0.6 wt.% SiCnp. The DTG curves show that as the SiCnp content increases, the maximum degradation rate of the PP/SiCnp occurs at a higher temperature. Given the presence of hydroxide groups in the PP chains, the numerous oxygen groups included within the SiCnp's backbone could establish strong hydrogen bonds. The SiCnp and PP matrix form stronger hydrogen bonds, which means that the SiCnp layers in the PP can prevent oxygen from penetrating and heat from transferring through the nanocomposites when they are heated. Therefore, the host polymer matrix's thermal stability might be significantly improved by a microscopic amount of SiCnp.

The essential temperature for applying pressure and the temperature ramps for hot compaction can be determined from the data shown above. These parameters are important for a homogeneous diffusion process, maximum densification of the NRAMCs, and good mechanical behavior.

The changes in hardness and elastic modulus in figure 10a and 10b. Both composites exhibited an improvement in mechanical characteristics within the diffusion zone, according to the data. There was a shift in hardness from 0.4 GPa to 0.7 GPa and an elastic modulus from 70 GPa to 140 GPa. One possible explanation for the enhanced mechanical qualities could be the incorporation of SiCnp into the aluminum matrix, which imparts hardness and stiffness to the composite. Hence, a future paper will discuss the scenario where the manufactured NRAMCs are anticipated to exhibit good mechanical behavior as a composite. Recrystallization near the nano reinforcement and metallic matrix could account for the diffusion zone's enhanced mechanical characteristics. Another possible explanation is the presence of crystalline contacts at the interface between the matrix and the reinforcement, which enable the transmission of load. Reports of declining qualities with increasing SiCnp concentrations have prompted the speculation that the mechanisms by which SiCnp and the metallic matrix reinforce one other remain a mystery. However, the results are very encouraging for using the sandwich approach to make nanocomposites with metal matrices.

Conclusion

The possibility of using PP to incorporate SiCnp into MMCs was explored in this study. Their appropriateness for this application was confirmed by the effective synthesis and thorough characterization of SiCnp. Results showed that the nano reinforcement had a substantial impact on the polymer, and the efficient dispersion of SiCnp into the PP matrix improved mechanical characteristics. The mechanical properties are reduced at high SiCnp levels because of agglomeration problems and/or low dispersion energies, which leads to poor distribution of SiCnp within the polymeric matrix, according to the dispersion quantification data. The production of me-

tallic nanocomposites was further helped by the PP reinforced with SiCnp, which showed good thermal degradation capability. The results of the nanoindentation experiments on the NRAMCs show that PP can be a good vehicle for adding nano reinforcements to metallic matrix, which bodes well for the future of advanced composites in many fields.

Acknowledgements

None.

Conflict of Interest

None.

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