



Characterization and Analysis of Carbon Fiber and Nano hBN Reinforced Hybrid Aluminium Metal Matrix Composites by Conventional Sintering

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Keywords

Hybrid composite
Nano composite
AlMMCs
Hexagonal Boron Nitride
Powder metallurgy

Abstract

As mono composites focus solely on improving one property at a time, the significance of hybrid composites grows day by day. The incorporation of carbon fiber (CF) and nano hexagonal boron nitride (hBN) particles as reinforcement for Aluminium has gained significant popularity because of their superior properties. In this work, the AA 7050 is reinforced with carbon fiber and nano hBN and fabricated by powder metallurgy method. To achieve an effective nanoparticle distribution in the matrix, premixing of the particles was done. The reinforcements were effectively dispersed by ball milling, and the composite was created using a traditional powder metallurgy. Dispersion of the particles in the matrix was analyzed by optical microscope. The effect of adding reinforcement to the matrix was investigated using properties such as micro hardness and compression test, and wear characterization. A significant increase in mechanical and wear properties was achieved for the combination of 0.25 wt. % carbon fiber and 0.5 wt. % hBN addition due to the uniform dispersion of nano particles along with the carbon fiber presence. The microhardness, compressive strength and wear rate is improved by 33%, 66% and 54 % respectively than the bare alloy. This study provides insight into the importance of hybrid Aluminium nano composites for high strength applications.

Research Article

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1. Introduction

The exceptional specific strength, specific stiffness, improved hardness, strong wear behaviour, and better endurance life of hybrid composites, which comprise two or more reinforcements, make them an unavoidable replacement for the commonly used materials [1]. The main challenge in the development of hybrid composites is the selection of the most effective combinations of reinforcements [2, 3]. When compared to conventional composites, hybrid composites offer distinctive properties that highlight various design requirements in an affordable manner. Better impact and fatigue resistance, reduced weight and cost, and balanced strength and stiffness are the key characteristics that distinguish hybrid composites. Furthermore, the majority of studies demonstrate that by negotiating certain other aspects, single particle reinforcement can improve any one of the properties [4].

In order to enhance the composite characteristics, a huge amount of research is being done on the production

of Aluminium Metal Matrix Composites (AlMMCs). AlMMCs and other light-metal composites demonstrate exceptional performance and remarkable characteristics in severe circumstances. Thermal stability at elevated temperatures is a requirement for the use of Al based Alloys in the aerospace and automotive industries. Even yet, there is still considerable work to be done to address the drawbacks of Al and its alloys under some severe circumstances, such as low strength, hardness, low corrosion resistance and poor tribological performance. Studies claim that, Aluminium alloys undergo a significant decrease in strength over 150°C, which is followed by a catastrophic softening as the temperature rises [5, 6].

To balance the ductility and strength of AlMMCs, numerous attempts have been made to incorporate different reinforcement particles into the composite. Al and its alloys are strengthened with fibers, whiskers, or ceramic particles to enhance their tribological and mechanical properties. Reinforcements such as Al₂O₃, SiC, TiO₂, Graphene, CNT and MoS₂, are frequently

employed [7–9]. Particulate reinforcements are a suitable choice due to its low cost, light weight, isotropic properties and easiness in manufacturing that is similar to that of conventional materials. The properties of AA 7075 reinforced with silicon carbide particles have gotten better mechanical properties [10]. Mechanical and microstructure properties of Al reinforced with Multi Walled Carbon Nano Tubes composites were studied and undesirable milling conditions weaken the mechanical properties of composites [11]. Hence choosing the finest milling conditions is a major challenge. Fiber reinforced materials enhance strength and stiffness at room temperature or at high temperatures, forming a robust composite, as opposed to strengthening the continuous phase [12].

The most frequently employed Aluminium alloys for structural applications are those of grade 7xxx, which have zinc as the main alloying element. The structural components of 50% of Boeing 787s and 25% of A380 Airbus are made of 7xxx alloys. Although having high thermal and electrical conductivities, excellent toughness, and other characteristics, AA 7050 is limited by its weak mechanical and tribological characteristics at extreme temperatures. AlMMCs can benefit from the addition of two or more reinforcements in addition to improved mechanical, metallurgical, and thermal properties [13, 14]. Although selecting the right reinforcement has a big influence on the process of making hybrid metal matrix composites, the main objective of the research is to identify the most effective ways to combine various reinforcements. Some of the primary concerns of the researchers are the various characteristics of reinforcements, including their high thermal conductivity, high strength-to-weight ratio, low density, better interfacial bonding, and resistance to chemicals, wear, and corrosion.

Ensuring characteristics of hBN nano powder are expected to overcome the limitations of the high-strength alloy, AA7050. In addition, hBN nano powder exhibits excellent thermal stability, wide aspect ratio, high strength, and high Young’s modulus, all of which make it beneficial for high-temperature applications. Because of its enhanced wear resistance and non-toxic nature, workability has been improved. The enhanced wear resistance and self-lubricating properties of reinforced hBN have been attributed to its lubricating properties. Because hBN is resistant to thermal shock, internal damage in the metal matrix produced by thermal shocks can be prevented [15, 16]. Studies claim that incorporation of hBN particles enhances the tribological properties [17, 18]. ZrCP and carbon fibers (CF) were reinforced to AA2024 using the spark plasma sintering technique. Multiscale and hybrid reinforcements can be added to AlMMCs to increase both their strength and ductility [19, 20].

The homogeneous dispersion of reinforcing particles, wettability problems, porosity, undesired chemical reactions, and creation of clusters or agglomerations are among the difficulties associated with the stir-casting technique. Using the stir-casting process, CNT and Al₂O₃ were added to AA 6061. Agglomerations were seen to arise at increasing concentrations, and it was challenging to make the composites at larger volumes [21]. The

minimal damage to the reinforcing particle during the casting process can be reduced by powder metallurgy [22, 23]. The most popular type of fabrication is powder metallurgy because of its nearly net shaped products, high strength, little scrap loss, homogeneous particle dispersion, and low propensity for agglomeration [24].

In this paper, the carbon fiber and nano hBN particles are added into the AA7050 through the powder metallurgy. Conventional sintering processing is employed to sinter the composites. The metallurgical, mechanical and tribological properties of the hybrid composite were studied.

2. Methods and materials

AA7050 gas atomized powder with a purity of 97%, density of 2.96 g/cm³, and a particle size of 5 μm is used as the base matrix in this work. The reinforcements chosen were carbon fibers and nano hexagonal boron nitride (hBN). Nano hBN powder with a grain size of 70 nm, purity of 99.8%, and a density of 2.1 g/cm³ is used as a reinforcement. The second reinforcement material is carbon fiber with a diameter of 6 μm and a density of 1.8 g/cm³. Based on the preliminary experiments, the carbon fiber weight percentage is taken as constant due to clustering of the carbon fiber while ball milling at higher concentrations. The weight percentage of carbon fiber in the investigation was set at 0.25 weight percentage, while the nano hBN varied between 0 to 2 weight percentages. The different weight percentages of AA 7050, hBN and carbon fiber were taken for the composite preparation as shown in Table 1.

Table 1. Weight percentage of different composite samples

Sample name	AA 7050	Carbon fiber	hBN
AA	100	0	0
AA0	99.75	0.25	0
AA25	99.5	0.25	0.25
AA50	99.25	0.25	0.5
AA75	99	0.25	0.75
AA100	98.75	0.25	1
AA150	98.25	0.25	1.5
AA200	97.75	0.25	2

2.1 Ball milling and Premixing

Ball milling and premixing were used to distribute the reinforcements throughout the AA7050 matrix. Figure 1 depicts the schematic representation of the powder preparation and processing procedure. At first, the materials for the matrix and reinforcements were chosen based on the weight percentages of each sample. After placing the hBN particles in the beaker containing the ethanol solution, they were subjected to ultrasonication at a frequency of 20 KHz for 30 minutes. Afterwards, carbon fibers were added, and the ultrasonication procedure was then continued. Following the ultrasonication procedure, AA 7050 were added to the mixture. After that magnetic stirring was carried out for

the next 30 minutes. To ensure an even distribution of particles, the matrix and reinforcing materials were combined, and magnetic and mechanical stirring were performed. Following the mechanical stirring procedure, the mixtures were dried at 80°C in an oven until all of the ethanol had evaporated.

The materials were ball milled at room temperature using a planetary ball mill following the premixing procedure. Two 250 ml jars are used for the study. The

jars and balls were made by tungsten carbide. The powders were milled for 4 hours, with 10-minute breaks given every hour to prevent the particles from adhesion. Methanol is utilized as the process control agent. The speed was adjusted to 200 rpm and the ball to powder ratio was selected at 4:1. Following the ball milling procedure, the samples were dried and packaged.

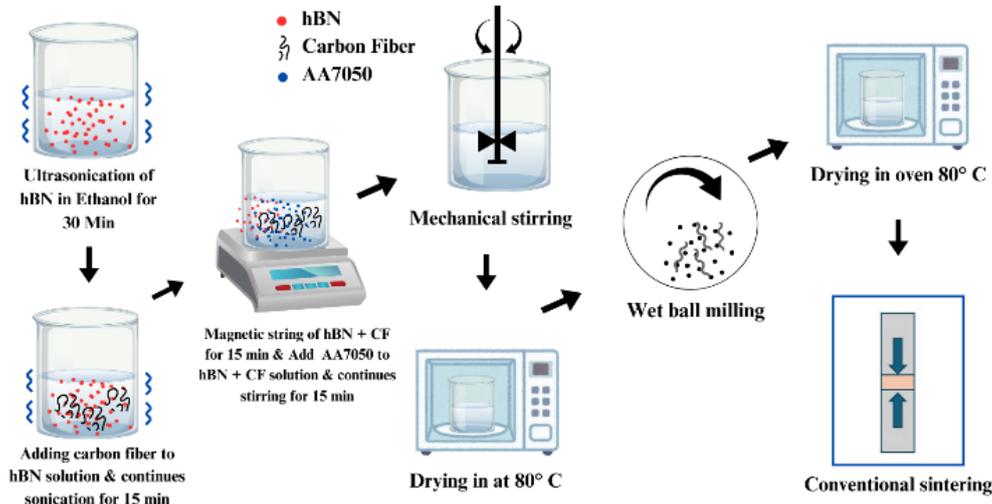


Figure 1. Schematic representation of hybrid composite fabrication

2.2 Compaction and sintering

After the premixing and ball milling, the powders were subjected to the conventional sintering process. The powders were cold compacted at 250 MPa, under a load of 2 kN. The powders were compacted at a die size of 12 mm diameter and a length of 24 mm. Zinc stearate was used as the lubricant in the compacting process to avoid the particles from sticking to the die. After the compacting process, the green compact was put in the furnace for the sintering process. The sintering was done at a temperature of 500°C for 1 hour under the argon atmosphere. After sintering, the samples were furnace cooled.

The sintered samples were subjected to different tests as per the standards. The microstructural analysis was done using the optical microscope, microhardness was measured by Vickers hardness tester, compression test was done using a Universal testing machine and wear analysis was conducted by a pin on disc apparatus.

3. Results and discussions

The theoretical density of the composite is measured by rule of mixtures and Archimedes principle is used to determine experimental density of the nano composite. The same samples was measured before and after immersing the samples in water. The equation for the same is shown in equation 1. Where M_1 is the composite's mass in air, M_2 is the nanocomposite's mass in distilled water, and ρ_w is the distilled water's density, which is equal to 1 g/cm³.

$$Density\ of\ composite,\ \rho = \frac{M_1}{M_1 - M_2} * \rho_w \quad (1)$$

The hybrid nanocomposite samples undergone XRD analysis and the results were plotted in Figure 2. The XRD patterns show that no more crystalline phases were formed during the manufacturing of the composite, and it's possible that certain phases were not detected due to machine constraints.

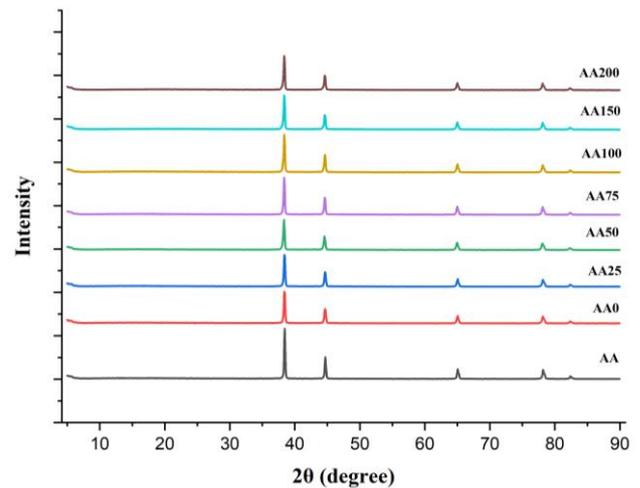


Figure 2. XRD of the composites at different wt. %

An inverted metallurgical microscope with a magnification range of 50X to 800X was used to analyze the microstructure of the samples using De-winter Material software. The samples were fine grinded and polished using 240, 400, 600, and 1000 grit emery sheets and velvet fabric. Keller's reagent is used to etch the polished surface. The grain structure was extracted from the composites by etching them for 10 seconds. Figure 3 shows the microstructural images of the composites at

200X magnification. The carbon fibers and hBN particles were clearly visible in the images. It was observed that above 0.5 wt.% of hBN addition into the AA 7050+ 0.25 wt.% CF leads to the clustering of nano particles. This clustering of the hBN nano particles in the hybrid composite may adversely affect the mechanical properties. Due to the temperature gradient in the conventional sintering process leads to the agglomeration of the nano particles. Along with that, porosity level of the hybrid composites was also observed to increase with the increase in the hBN concentration.

The mechanical properties of the hybrid composite material are significantly influenced by porosity in the composites. They serve as pre-existing cracking sites where crack propagation and initiation take place. Research findings indicate that prolonged ball milling can lead to structural alterations that may deteriorate the

properties of the ceramic particles [25]. As stir casting process is not recommended for reinforcing nano and fiber particles, sintering process is commonly adopted. High porosity levels during the sintering process are caused by a thermal mismatch among the matrix and reinforcing components. The increase in clustering may be due to the uneven distribution of the nano hBN particles in the matrix. For a particular level, the premixing process helps to get uniform dispersion [26]. From Figure 3, the grain boundaries were observed to be clearly visible at lower concentrations due to the slow solidification rate of the composite samples. These resulted in the formation of fine grains up to 0.5 wt.% hBN addition and higher concentrations resulted in coarse grains which reduces the properties of the composites.

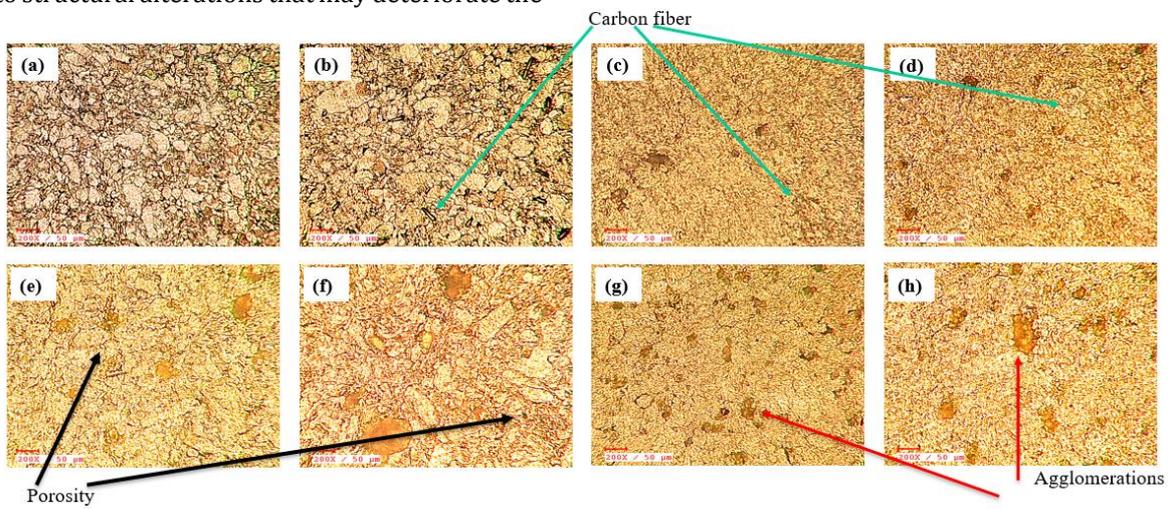


Figure 3. Optical microstructure of the composites at different wt. % (a) AA (b) AA0 (c) AA25 (d) AA50 (e) AA75 (f) AA100 (g) AA150 (h) AA200

Microhardness of hybrid nano composite samples were measured according to ASTM E384-16 standard, and employing a Vickers hardness tester with a 15 second dwell period and a testing load of 0.1 kgf. The average microhardness of the 5 different positions of each sample were reported in Figure 4. It was observed that the microhardness of all the samples is more than that of the base alloy, AA 7050. The addition of carbon fiber enhances the hardness of the samples due to the fiber nature of the reinforcement. The incorporation of hBN along with fixed carbon fiber concentration initially resulted in increased microhardness till 0.5 wt % hBN addition and then decreased. The bare alloy has the lowest microhardness value (45 HV) and the highest microhardness value (60 HV) is reported at 0.25 wt.% Cf and 0.5 wt.% hBN addition. Hence a maximum percentage improvement of 33.33 % in microhardness value is reported for the hybrid composite. While adding hBN nano particles, along with carbon fiber also leads to the improvement of microhardness to a certain extent till 0.5 wt.% hBN concentration and then decreases with an increase in hBN concentration. The increase in microhardness of the hybrid composite is mainly due to the presence of carbon fiber and homogeneous dispersion of ceramic reinforcing particles which efficiently restricts the grain movement in the alloy

matrix material [27]. This resulted in minimizing grain deformation and enhancing the mechanical properties of the composites.

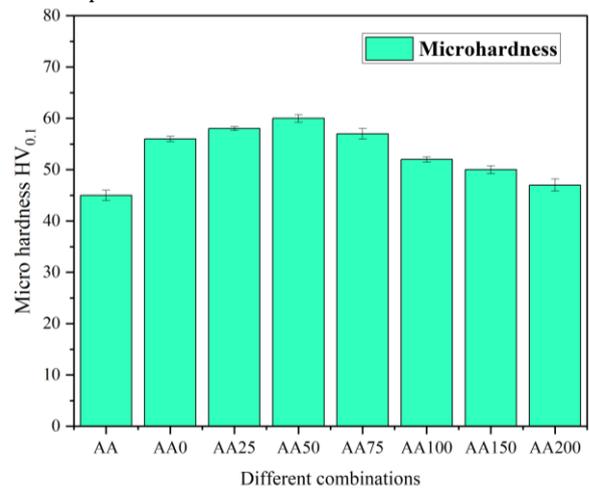


Figure 4. Variation in microhardness of the hybrid nano composites at different combinations

The reduction in the microhardness value, above 0.5 wt. % hBN addition is due to particle agglomeration, porosity, and uneven distribution of reinforcement particles within the matrix, which is visible in Figure 3. Since hBN is a soft phase material, it is difficult to embed

large amounts of nano hBN in the matrix. The increase in agglomeration at higher hBN concentrations is mainly due to the poor sinterability of the material, which adversely affects the hardness value [28].

The compression test of each nano hybrid composite sample was tested according to ASTM E9 and the average value is plotted in Figure 5. For the compression test, the cylindrical specimens having a length to diameter ratio of less than two are prepared. Universal testing equipment with maximum capacity of 1000 kN was employed to assess the compression strength. The variation of compressive strength of the hybrid nano composites is shown in Figure 5. From Figure 5, the compression strength of the carbon fiber composite shows a maximum value of 85 MPa and the bare alloy has a strength of 48 MPa. This is mainly due to the inclusion of fiber particles in the matrix, which strengthens the matrix phase. This may prevent crack propagation and hence boost the strength of the developed composite [29]. Up to 0.5 wt.% hBN addition, there is a small drop in the compressive strength than the peak value. Further addition of the hBN particles into the matrix along with carbon fiber resulted in a drastic drop in the strength of the composites. The decrease in compressive strength at higher concentrations is due to the agglomeration and soft nature of the hBN nano particles, which is visible in microstructural analysis (Figure 3). This agglomeration on the particle boundary leads to the formation of void spaces, and results in higher porosity at higher hBN concentrations.

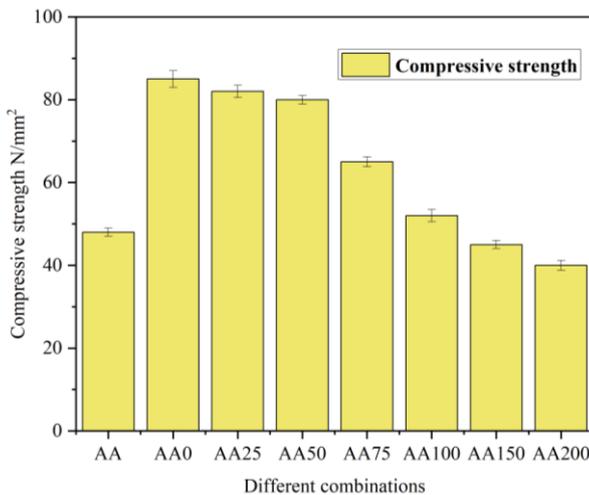


Figure 5. Variation of compressive strength of the hybrid nano composites at different combinations

The wear rate of hybrid nano composite was assessed at room temperature using the ASTM G99 standard. A pin on disc with a maximum speed of 2000 rpm is used for wear analysis. The sliding distance of 120 mm, sliding velocity of 1.2 m/s, sliding time of 10 minutes, speed of 600 rpm, and worn track diameter of 40 mm were held constant throughout the experiment. Figure 6 displays the variation wear rate under different load conditions such as 10 N, 25 N and 40 N. It was found that the composite wear rate was lowest at 0.25 weight percentage CF and 0.5 weight percentage hBN addition. The addition of carbon fiber alone into the matrix enhances the wear behaviour. The further addition of hBN addition along with carbon fiber reduces the

composite wear rate due to the solid lubrication property of the hBN. For higher hBN concentrations, the wear rate is shown to be increased till 0.5 wt.% addition due to the even dispersion of the nano particles. Beyond 0.5 wt.% hBN addition, the composite wear rate increased due to agglomeration, uneven distribution and higher porosity, which is evidently visible from microstructural analysis as shown in Figure 3. The reinforced material continues to perform significantly better than base alloy, even when the wear loss due to particle pull-out increases with load [30]. The self-lubricating properties of hBN and its resistance to plastic deformation primarily contribute to the improvement in wear resistance.

In general, the single reinforcement of the particles in the Aluminium metal matrix only improves one property of the composite by sacrificing the other properties. This study focused on developing a hybrid nano composite by reinforcing carbon fiber and nano hBN particles to obtain improved mechanical and tribological properties which is essential for high strength applications. In this study, carbon fiber addition enhances the mechanical properties, while the hBN particles improve the wear behaviour. As per the research, the optimum weight percentage for getting notable mechanical and tribological properties was at 0.25 wt. % CF and 0.5 wt.% hBN addition.

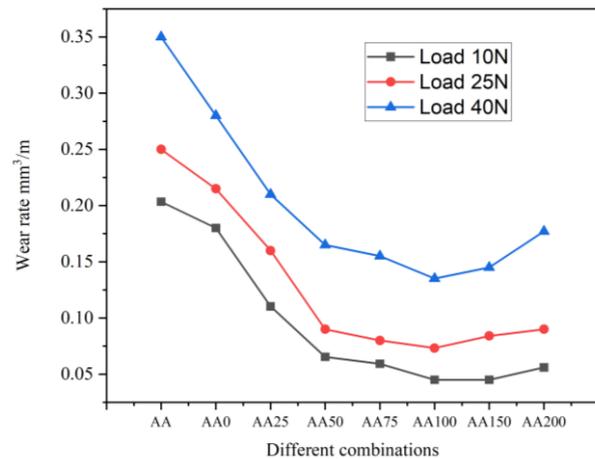


Figure 6. Hybrid nano composite wear rate at different combinations

4. Conclusion

In this work a detailed microstructural, mechanical and wear analysis of carbon fiber and nano hBN reinforced hybrid Aluminium metal matrix composite by conventional sintering was studied. Hybrid nano composite testing and characterization were performed as per ASTM standards. Mechanical properties such as microhardness and compressive strength, and wear analysis for different concentrations of nano hBN with 0.25 wt.% carbon fiber reinforced hybrid Aluminium metal matrix composite was investigated in detail. The major conclusions are listed below:

- A Significant increase in mechanical and wear properties was achieved for the combination of 0.25 wt.% carbon fiber and 0.5 wt. % hBN addition due to the uniform dispersion of the nano particles along with the fiber content.

- The grain boundaries were clearly observed at lower hBN concentrations due to the slow solidification rate of the composite and it resulted to the formation of fine grains upto 0.5 wt.% hBN addition.
- The addition of the reinforcements initially results in an increase in microhardness till 0.25 wt. % CF and 0.5 wt.% hBN. The increase in microhardness of the hybrid composite is mainly due to the homogeneous dispersion of ceramic reinforcing particles which efficiently restricts the grain movement in the alloy matrix material.
- The small addition of hBN along with 0.25 wt.% CF till 0.5 wt.% hBN gives almost the same compressive strength compared with carbon fiber alone.
- The decrease in the mechanical properties above 0.5 wt. % hBN addition is due to particle agglomeration, porosity, and uneven reinforcement particle distribution, which is visible from microstructure analysis.
- The maximum improvement of 33 % microhardness and 66% compressive strength were reported for the developed hybrid composite.
- It was found that composite wear rate was lowest at 0.25 weight percentage CF and 0.5 weight percentage hBN addition due to the presence of carbon fiber along with the solid lubrication property of the hBN
- This study provides insightful information on the importance of hybrid carbon fiber-hBN Aluminium nano composites for high strength applications.

Author contributions

Nice Menachery: Conceptualization, Writing - original draft, Writing - review & editing. **B. Deepanraj:** Writing - review & editing. **Shijo Thomas:** Supervision, Writing - review & editing.

Conflicts of interest

The authors declare no conflicts of interest.

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