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INVESTIGATION OF MECHANICAL AND TRIBOLOGICAL PROPERTIES OF ALUMINIUM HYBRID NANOCOMPOSITE PRODUCED BY NOVEL STIR-ULTRASONIC-SQUEEZE CASTING METHOD

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Abstract

This work uses a novel stir-ultrasonic-squeeze casting technique to fabricate hybrid nanocomposites based on aluminium alloy 6061 (AA6061). Owing to its excellent strength, ability to withstand corrosion, and formability as a matrix material, AA6061 was chosen. The process involves adding boron carbide (B₄C) & graphite and (Gr) nanoparticles (n_p) to the aluminium melt, followed by mechanical stirring, ultrasonic agitation, and squeeze casting to produce the sound casting. The weight percentages of (B4C and Gr) n_p were taken at a constant 2wt%. The mechanical and tribological properties of these hybrid metal matrix nanocomposites (HMMNCs) are examined in this study. A Brinell hardness number and ultimate tensile strength (UTS) in MPa examined are 66.9 and 197.01 with a 27.02% and 4.06% increment for (2wt%B4C+2wt%Gr). A pin-on-disk (POD) tribometer was used to analyze the dry sliding wear of the composite. Significantly, compared to AA6061 (as cast), the wear characteristics show a lower average coefficient of friction and wear rate for 2wt% HMMNCs. For all loads and sliding velocities, a better increase is thus seen, highlighting the better mechanical and tribological performance of 2wt% B4C with 2wt% Gr.

Keywords:

Aluminium, hybrid nanocomposites, wear, stir casting, ultrasonic casting, squeeze casting, COF

1 INTRODUCTION

Aluminium alloys are extensively utilized in various industries, including automotive and aerospace, due to their lightweight and high-strength properties. The demand for materials with a high strength-to-weight ratio has driven research towards metal matrix composites (MMCs), particularly those with aluminium matrices. These composites are known for their low cost, ease of fabrication, low density, and excellent mechanical properties. Aluminium matrix composites have demonstrated significant benefits in engine applications, reducing overall weight, fuel consumption, and emissions in vehicles. Major brands like General Motors, Toyota and Boeing have incorporated aluminium matrix composites in select products for enhanced performance.[Abúndez 2016] [Rohatgi 1998] [Aigbodion 2019]. MMCs are widely utilized for its exceptional qualities: low density, high specific strength, corrosion resistance, stiffness, stability at high temperatures, reduced part weight, low thermal shock, and enhanced mechanical properties [Babaremu 2019] [Pandey 2018].

The focus has shifted to the currently fascinating 6XXX alloys, popular for medium strength, formability, weldability, wear resistance, corrosion resistance, and cost-effectiveness. Notably, AA6061, within the 6XXX range, offers superior wettability, castability, and customizable specific strength through optimal heat treatment. This makes it widely applicable in automotive ancillary components, this alloy is often enhanced with reinforcements like SiC, TiO2, Al2O3, B4C, Si3N4, AlN, TiC, ensuring improved compressive strength and wear resistance[Nallusamy 2022]. Diverse reinforcements such as Al, Ni, Cr, Gr, TiB2, ZrB₂, CNTs, B₄C, GFRP, and metal alloys enhance elevated temperature thermal stability, corrosion resistance, wear resistance, fracture toughness, tensile strength etc [Badheka 2018]. Ceramic reinforcement selection considers factors like elastic modulus, tensile strength, density, size, and shape, melting temperature, thermal stability, and coefficient of thermal expansion, compatibility, and cost. For fiber-reinforced MMCs, parameters like fiber orientation, aspect ratio, mechanical properties of fibers and matrix, and the bond nature between them significantly influence the selection [Aravindan 2015]. As per the exhausted literature search of (Al-MMC) based on single reinforcements reported in the last half decades B4C [Shrivastava 2019][Badheka 2018][Badheka 2016] SiC[Dinaharan 2016][Bodukuri 2016][Hamdollahzadeh 2015], TiC [García-Vázquez
2016],[Dinaharan 2014], Al2O₃,[García-Vázquez Al₂O₃,[García-Vázquez 2016],[Jamwal 2020], SiO² [Selvakumar 2017],[Joyson 2016].

There is a tremendous need for aluminium alloys with enhanced mechanical and wear-resistant traits, especially for automotive components like drive shafts and disc brakes. Graphite nano reinforcements

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strengthen the matrix material, while aluminium metal matrix hybrid nanocomposites (AMMHNCs) aim to
surpass conventional AMMCs. Secondary conventional reinforcements like B₄C, SiC, Al_2O_3 , and FeTiO₃ enhance performance. AMMHNCs use multiple reinforcements, providing a broad range of options to enhance the base matrix's mechanical properties [Rajesh 2016], [Zhou 2020].Researchers commonly utilized graphite, molybdenum disulphide, and silicon carbide in combination with an aluminium base matrix for AHMMC fabrication. The AMMHNCs demonstrated improved mechanical performance compared to AMMNCs with a single reinforcement IN, G. S. Kumar 2020],[Jamwal 2020]. Machining composites is challenging due to abrasive hard reinforcement particles like boron carbide and silicon carbide in the base matrix [Saini 2023].

Researchers are creating cost-effective hybrid metal matrix composites (HMMCs) using graphite fibers and particles- a high-strength, low-density material with various solid lubricant particles[Jeon 2014][Reddy 2019][Lagisetti 2022].Increasing graphite content in composites reduces surface roughness providing selflubricating properties, lowering fuel consumption, and minimizing energy expenditure. These aluminiumgraphite MMCs, used in automotive bearings, exhibit low friction coefficients, reduced wear rates, and excellent anti-seizing properties. Graphite's low density and self-lubrication make it a suitable reinforcement, offering thermal stability to aluminium metal matrix composites (AMMCs)[Lagisetti 2022][Gnaneswaran 2022][Christy, 2019][Lisheng 2020].Ceramic particles (SiC, B₄C, Al₂O₃, and TiC) enhance tribological properties in MMCs. B₄C, with a density of 2.52 g/cm³, stands out as the third hardest substance, offering excellent wear resistance. Its high melting point (2445°C) enhances thermal properties and resistance to various chemicals [Shrivastava 2019][Viswanatha 2013]. Amongst different ceramic particles, graphite is the most suitable reinforcement which possesses low density, and self-lubricating properties, in a result this improved the machinability of aluminium. It can easily be incorporated into the melt using cheap and widely available [Gupta 2022][Pai 2015].

AMMNCs are produced through various methods like stir casting (liquid metallurgy), powder metallurgy (solid metallurgy), high-energy ball milling, and spray deposition. Stir casting is popular for its ease and costeffectiveness but faces issues like uneven reinforcement dispersion. Researchers attempt to address this through combinations like stir-ultrasonic casting, but porosity remains a challenge. Squeeze casting is explored to counter porosity [Verma 2023][Pandey 2017].

This study focuses on AHMMNC manufacturing, utilizing a combination of stir-ultrasonic-squeeze casting for uniform dispersion and minimal porosity. The approach involves 2wt%B4C-n^p as primary reinforcement and 2wt%Gr-n^p as secondary reinforcement. The liquid metallurgy route is chosen for economic mass production, aiming to analyze the processed nanocomposites' mechanical and tribological properties. The use of a novel stir-ultrasonic-squeeze casting method represents a significant advancement in the fabrication of aluminium hybrid nanocomposites. This multi-step process combines the benefits of mechanical stirring, ultrasonic agitation, and squeeze casting to achieve a uniform dispersion of nanoparticles and reduce porosity in the composites. Each step contributes uniquely. By integrating ultrasonic agitation with mechanical stirring and squeeze casting, the study expects to achieve a more uniform distribution of B4C and Gr nanoparticles within the AA6061 matrix. This enhanced dispersion leads to significant improvements in mechanical and tribological properties. Reducing porosity is critical for achieving higher strength and better wear resistance.

2 EXPERIMENTAL

This work mainly focused on the fabrication and assessment of mechanical and tribological properties of AMMHNCs by utilizing and combining different
approaches i.e. stir-ultrasonic-squeeze casting stir-ultrasonic-squeeze technique. The subsequent subsections provide further details about the experimentations.

2.1 Selection of Raw Materials and Procurement

AA6061 has superior tensile strength, superior corrosion resistance coupled with superior weldability and workability and is ideally suitable for automotive applications. Thus, for this research experimental work aluminium alloy 6061 is selected. AA6061 is a precipitation-hardening alloy, containing magnesium and silicon as its major alloying elements. AA6061 was procured from Markandey Enterprises, Bhopal, India. The composition of the as-received aluminium alloy 6061 was confirmed from the spectroscopy analysis test i.e. Optical Emission Spectroscopy (OES) as per ASTM standard ASTM-E- 1251:2017 and the elemental composition of the alloy (as shown in Tab. 1). The emission spectroscopy technique used for the elemental composition analysis of bulk samples is very reliable due to its complexity. The sample was prepared from as received material through a conventional machining procedure as per ASTM dimensions i.e. 20 mm in diameter and 20 mm in length (refer Fig. 1). The matrix material AA6061 is used in as received condition. The chemical composition of the AA6061 as supplied by the manufacturer is shown in Tab. 1.

Fig. 1: Shows OES spectro testing (a) Test specimen (b) sample mounted on OES testing (c) Atomic emission spark method.

2.2 Reinforcement particles

To develop AHMMNC, two types of reinforcement particles B4C nanoparticle (purity 99.9%, APS 50nm) and Gr nanoparticle (purity 99.9%, APS 100nm) were used. Both the reinforcement particles were procured from Nano Research Lab, Jamshedpur, India.

2.3 Manufacturing of novel cast AMMHNCs

The stir casting equipment used had an electrical resistance furnace consisting of a bottom pouring, mechanical retractable stirrer facility, capable of a maximum temperature of 900°C. The setup was manufactured and supplied (M/S) by Swam Equip, Chennai, Tamilnadu, India. It has two separate modules ultrasonic arrangements and hydraulic squeeze die casting (refer Fig. 2). The ultrasonication probe is of high melting material (Ti6-Al-4V) composition. The inert gases mixture of argon (Ar) and Sulphur hexafluoride (SF_6) was purged within the melting furnace in a ratio of 3:1. It was used during the mixing and pouring of slurry to prevent and make it oxidation resistant atmosphere inside the furnace melt. AA6061 was used as the matrix material, and 1.5 kg of the alloy were cut into small pieces and placed into a graphite-coated stainless steel (SS) crucible. Small quantity of magnesium was added to improve the wettability of the reinforced phase into the matrix.[Das 2012]. The material was melted in a graphite-coated crucible which is fixed with SS retort. The aluminium alloy was heated up to 750ºC. The preheating of nanoparticles was carried out at 250 ºC separately in a muffle furnace. After melting the matrix material, the preheated 2% wt. (B₄C and Gr) n_p were added to the melt in the encapsulation with aluminium

2.4 Measurement of physical and mechanical properties

2.4.1 Density and porosity assessment of hybrid nanocomposites

The Archimedes method was used to determine the experimental density and porosity of hybrid nanocomposites. To measure the density and porosity, a sample size of 1 cm^3 dimensions (approx.) was machined from hybrid nanocomposites through a conventional machining procedure followed by ultrasonic cleaning. The samples were first weighed in air and then in distilled water by using the following formula:

$$
\sigma_{experimental} = \left(\frac{m_{air}}{m_{air} - m_{water}}\right) \times \rho_{water}
$$
\n
$$
\varphi = 1 - \left(\frac{\rho_{experimental}}{\rho_{therotical}}\right) \times 100
$$
\n(2)

Where m_{air}, and m_{water} is the mass of AMMHNCs in dry and suspended conditions, respectively, and ρwater, is the density of water. The theoretical density was determined using the mixture rule.

2.4.2 Hardness measurement

Brinell hardness testing machine, (Make: INNOVATEST, Model no. VERZUS 750U), was utilized

foil in capsule form. Instantaneously after the addition of nanoparticles, mechanical stirring was carried out at 450 rpm for 6 minutes. The melt was then agitated by ultrasonic vibration at a defined frequency of 20 kHz for 2 minutes. After completing both steps i.e. mechanical stirring and ultrasonication, the slurry melt was passed through the bottom preheated pathway of the stir casting setup into the squeeze casting mould. The mould was preheated before transferring the slurry. The squeeze casting setup used a piston operated at 30 MPa pressure for 15s to squeeze the slurry till solidification was completed. The solidified casting was taken out of the squeeze casting setup and allowed to cool to room temperature before further processing the material. Finally, specimens are cut from the squeeze cast as per ASTM standards for further examination.

Fig. 2: The gravity pouring stir-ultrasonic-squeeze casting set-up

by ASTM E10-00 standards. A 2.5 mm-diameter hardened steel ball spherical in shape was used as the indenter, applying a force of 31.25 kg-F for a dwell time of 15 seconds. Samples were obtained from the top, middle, and bottom positions of cast specimens and subsequently polished. The specimens were sliced into dimensions of 10 mm thickness and 50 mm diameter**.**

2.4.3 Tensile measurement

The tensile test specimens of the nanocomposites were fabricated in compliance with ASTM standard E8 universal testing machine (model-INSTRON 8801K1403) was utilized to perform tensile tests at room temperature. The specimens were subjected to a tensile load at a crosshead speed of 0.5 mm/min. For reliable results, three samples of each composition were analyzed. Yield strength, ultimate tensile strength (UTS), Young's modulus, and percentage elongation are calculated from the tensile test data.

2.4.4 Wear test

The dry sliding wear experiments were done by ASTM G 99-17 standards using a pin-on-disk wear testing machine (Make: Ducom, and Model no. TL-20 Neo Series). The pin was machined to have a diameter of 10 mm and a length of 25 mm. An electronic weighing scale with a 0.0001 mg resolution was used to measure the weight. The EN-31 steel disc utilized in the Pin-on-Disc testing had a hardness of 62 HRc. Its dimensions were 165 mm in diameter and 8 mm in thickness. The wear rate of test specimens, measured in $×10⁻³$ mm3/m, is determined by measuring weight loss during sliding for three different applied loads (10, 20, and 30 N), sliding speeds (0.84, 1.26, and 1.68 m/s), and sliding distances held constant at 1500 mm and 40 mm from the disk center.

produced by a novel stir-ultrasonic-squeeze casting method reinforced with (B₄C and Gr) n_p in different weight proportions.

3.1 Physical properties of the AMMHNCs.

The theoretical, experimental porosity, and density of the AA6061 and Al-(2wt%B4C+2wt%Gr) AMMHNCs is shown in Tab 2. The density of AMMHNCs showed marginal changes at higher proportions, attributed to the inclusion of lightweight n-Gr particles. Specifically, for (2wt %B₄C+2wt%Gr) n_p and the density increased by 0.15%. The experimental density closely aligned with theoretical density, and AMMHNCs exhibited a favorable porosity range of 2.7% better than the (as cast) base unreinforced alloy (1%) due to the novel stir-ultrasonicsqueeze casting method.

3 RESULTS AND DISCUSSIONS

The major findings in this experimental work are to assessment of physical, mechanical i.e. (hardness & tensile), and wear characterization of AMMHNCs

Fig. 3: (a) Showing the variation of densities (b) porosity of AMMHNCs varying wt% of (B4C & Gr) np.

3.2 Brinell Hardness Test (BHN)

The BHN of AMMHNCs (2wt%B4C+2wt%Gr), increased by 27.2%, and as compared with unreinforced as-cast alloy. The average values of BHN are revealed in Tab. 3, and the variation of various hybrid nanocomposites is shown in Fig. 4.

3.3 Tensile measurement

An universal testing apparatus was to carry out tensile tests. The Young's modulus, UTS and elongation (in %) for AA6061 (As Cast) and AHMNC with added reinforcements of 2wt%B4C and 2wt%Gr are also determined.

When compared to the AA6061 (as cast), Young's modulus and UTS of the hybrid nanocomposites have significantly increased with the added reinforcement (B_4C) n_p and (Gr) n_p particles. The UTS of the AA6061 (as cast) is observed as 186.01 MPa and the UTS of the AHMMNC (2wt%B4C+2wt%Gr) is observed as 195.965 MPa with an increase of 5.351 %. The Young's modulus

of the AA6061 (as cast) is observed as 23.486 GPa and the Young's modulus of the AHMMNC (2wt%B4C+2wt%Gr) is observed as 24.021 GPa with an increase of 2.277 % (refer Tab 3). On the other hand, it is noticed that by addition of the reinforced phase of B4C and Gr n_p, the percentage elongation of the AHMMNC decreases (See in Fig. 8). The ductility of the nanocomposites is determined by the percentage elongation. Because of the injection of dislocations, the reinforced phase limits the alloy matrix's ductile qualities to some extent [Dabade 2021][Ammisetty 2022]. Smaller grains are then formed as a result, significantly decreasing the hybrid nanocomposite's ductility.

Fig. 5: (a) Shows the Stress vs. Strain graph (b) effect of AHNCs varying wt% of (B4C & Gr) np on yield Strength and young's modulus and (c) UTS and percentage elongation of AHNC.

3.4 COF and wear of hybrid nanocomposites.

The study investigates the frictional behavior of AA6061 and its hybrid nanocomposites with different wt% of B4C and Gr n^p under varying loads and sliding speeds. See in Fig. 6: illustrates the average coefficient of friction (COF) trends for different compositions. The avg. COF for AA6061 (As cast) of all materials increases with applied normal loads, but (2wt%B₄C+2wt%Gr) n_p shows a decrease. The incorporation of 2wt% B4C particles reduces the avg. COF, compared to the base alloy. (2wt%B4C+2wt%Gr) n^p exhibits a lower avg. COF compared to AA6061 (as cast) at varying loads and sliding speeds, with reductions of 47.05%, 34.5%, and 31.5% at 10N; 18.5%, 5.4%, and 20% at 20N; and 35.5%, 35.6%, and 27% at 30N.

Higher applied normal loads lead to an increase in COF values due to greater depth of penetration, but at extremely high loads, the face between (B₄C-Gr) n_p and matrix breaks down, causing a significant COF increase. The avg. COF decreases at higher sliding velocities due to the softening of the composite pin specimen and EN31 steel disc, attributed to frictional heat and flash temperature increase. Incorporation of Gr-n_p minimizes heat generation and further avg. COF reduction is observed in AMMHNCs due to the tribo-layer on the contact surface. The hexagonal structure of graphite reduces COF in HNCs. [Prasad Reddy 2019]. In conclusion, the study provides insights into the frictional behavior of the investigated materials, highlighting the influence of nanoparticle composition on COF under varying conditions.

3.5 The wear rate of pin materials.

Wear rates are higher in AA6061(as Cast) compared to AHMMNC under all conditions. (2wt%B4C+2wt%Gr)n^p composites exhibits a lower wear rate compared to AA6061 (as cast) at varying loads and sliding speeds, with reductions of 10.27%, 3.21%, and 13.84% at 10N; 34.98, 16.47 and 7.97% at 20N; and 21.15, 6.89%, and 6.84% at 30N. (refer Fig. 7) illustrates the variation of wear rate. Better wear resistance is observed in $2wt\%B_4C-n_p$ compared to the base material due to the

formation of a mechanically mixed layer (MML) with hard B₄C-n_p acting as a barrier and increasing nanocomposite hardness.

Wear rates increase with sliding velocity, leading to elevated temperatures at the pin-EN31 steel disc interface and inducing oxidation. Heavy heat dissipation affects the chemical structure of the pin material. Incorporation of Gr-n_p forms a stable lubricating layer, enhancing wear resistance compared to the base alloy. The dual-phase effect of $(B_4C\text{-}Gr)$ n_p contributes to a stronger MML on the contact surface.[Prasad Reddy 2019] [Baradeswaran 2013].

Fig. 6: (a), (b), and (c) shows the variation of avg. COF under varying loads for AHMMNC.

Fig. 7: (a), (b),& (c) Shows the variation of wear rate in (×10-3 mm³ /m) under varying loads for AHMMNC.

4 SUMMARY

This experimental study focuses on manufacturing AA6061 reinforced with hybrid (B₄C+Gr) n_p , produced through a novel stir-ultrasonic-squeeze casting process. The major conclusions drawn from the mechanical and tribological performance, as compared to AA6061 (as cast), are listed below:

• The density of the $(2wt%B_4C+2wt%Gr)$ n_p composites showed increment of 0.15%, attributed to the incorporation of B_4C -n_p particulates.

• The novel stir-ultrasonic-squeeze casting method is highly effective in producing aluminium hybrid matrix nanocomposites (AMMHNCs) with exceptional properties. Compared to the 1% in the unreinforced (as cast) alloy, AMMHNCs have higher porosity levels 2.7%. The method exhibits robustness and potential for producing high-quality, low-porosity composites, which enhances the overall performance of the AMMHNCs.

• Brinell hardness number (BHN) of AMMHNCs showed a substantial increase of 27.02% stabilizing at higher B_4C -n_p weight percentages, likely due to the inclusion of Gr-np.

 The developed hybrid nanocomposites exhibit enhanced tensile properties including higher elastic modulus, and UTS possibly due to reduced grain size ascribed by Hall Petch relationship.

 Dry sliding wear tests revealed a reduction in average coefficient of friction (COF) and wear rate for AMMHNCs compared to unreinforced AA6061 (as cast).

• (2wt%B₄C+2wt%Gr) n_p composition composites exhibited a lower average COF than AA6061 (as cast), attributed to the inclusion of lower-density nanoparticles compared to 2wt%B4C-np.

• Wear rate decreased for $(2wt%B_4C+2wt%Gr)$ n_p composite compared to AA6061 (as cast) due to higher B₄C-n_p contents.

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