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Development and Mechanical characterisation of multi-walled carbon nanotubes and hydroxyapatite reinforced ADC12 aluminium alloy hybrid metal matrix composite

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Abstract. Metal matrix composites represent an advanced class of materials with enhanced properties over conventional monolithic alloys. This study aims to investigate the mechanical characteristics of ADC12 aluminium alloy reinforced with multiwalled carbon nanotubes and hydroxyapatite under a systematic varied process condition using Taguchi optimisation approach. Four specimens of different composition and melt temperatures, and its mechanical properties were fabricated and evaluated for Impact strength, Rockwell hardness, Vickers micro hardness. The test results revealed that the specimen AL-232 absorbed the maximum impact energy of 2.13 J under Charpy V-notch impact test, highlighting optimal balance between reinforcements and metal matrix, maximizing toughness. Micro-Vickers hardness shows the highest hardness value of 157.5 HV is observed in specimen AL-213 and Rockwell hardness value of around 56HRB exhibited in specimen AL-312 and AL-213. This research aims to carry strategic process parameter optimisation along hybrid reinforcement composition to design and develop aluminium ADC12 based hybrid metal matrix composites.

1. Introduction

The development of metal matrix composites (MMCs) represents a significant advance in materials engineering. MMCs achieve a synergetic effect by combining reinforcements into metal matrix that increases the performance to be made suitable for specific applications. Aluminium alloys are appealing in metal matrix composites due to its low-density, allowing processing flexibility among various manufacturing facilities [1], [2]. Integration of multi-walled carbon nanotubes amid their higher individual mechanical properties have been reported of boosting the performance of various materials [3], [4], [5]. Hydroxyapatite is a

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bio ceramic with numerous applications of medical implants due to their excellence in biocompatibility. The primary obstacle faced during incorporation of nanoscale particles into a metallic matrix is achieving homogenous dispersion[6], which is challenging due to the high surface area to volume ratio property of both the reinforcements, promoting higher risk of particle agglomeration as compared to the required even distribution ideally, resulting in substantial reduction of the effective improvements of the composite by uneven load distribution[4], [7]. High pressure die casting with ultrasonic assistance was employed to reduce particle agglomeration, ensuring reinforcement distribution into aluminium vortex [8], [9], [10]. This study contributes to addressing a critical gap in the materials landscape by providing a deeper understanding of the characteristic improvement of the ADC12 aluminium alloy upon reinforcing with multiwalled carbon nanotubes and hydroxyapatite contributing to foundational understanding of composite materials.

2. Materials and Methods

2.1 Materials

2.1.1 ADC12 aluminium alloy

ADC12 aluminium alloy is widely used in die casting because to its excellent castability, mechanical properties and corrosion resistance, featuring a high silicon content up to 12% combined with copper, magnesium, zinc, iron providing excellent castability, mechanical strength due to its exceptional fluidity and minimal shrinkage during solidification[2], [11], [12]. It has a density of 2.74 g/cm^3 and 600 °C melting point[11]. The alloy's superior castability, strength, corrosion resistance and low cost, make it preferable for mass production of intricate die cast parts across industries[11], [13].

2.1.2 multi-walled carbon nanotubes

Multiwalled carbon nanotubes are cylindrical nanostructures of multiple concentric graphene tubes with diameters ranging from a few nanometres to tens of nanometres providing exceptional mechanical strength, with tensile strength reaching up to 10–60 GPa, with high stiffness, resilience, electrical conductivity, chemical stability and outstanding thermal conductivity making them suitable for reinforcing materials[4], [5], [6]. Multiwalled carbon nanotubes are used as reinforcements to enhance mechanical properties such as strength, stiffness, and fatigue resistance of the composite[4], [5]. The multiple layers in multiwalled carbon nanotubes provide improved toughness and durability, and functionalization of these surfaces can improve bonding with metal matrices[6], [7].

2.1.3 Hydroxyapatite

Hydroxyapatite is a calcium-based phosphate ceramic that closely mimics the inorganic phase found in human bone and teeth. Its excellent biocompatibility, inherent bioactivity, and strong osteoconductive properties makes it extensively applicable in biomedical uses such as bone grafts, bone implants, and coatings for improving bone integration[14], [15]. Hydroxyapatite incorporation into MMCs improves composite hardness and can suit applications where both structural performance and bioactivity are critical, such as in orthopaedic and dental implants[15], [16].

2.2 Fabrication

Initially the furnace, mould, stirrer, and ultrasonic probe are thoroughly cleaned and treated with a graphene coating to prevent damage from the molten metal. The furnace temperature is set according to the L9 orthogonal array and mould is preheated up to 400°C[9], [10], [17]. The multiwalled carbon nanotubes and hydroxyapatite are measured to the weight percentage of the metal according to the L9 orthogonal array and are fed into a powder feeder which preheats the powder to 350°C[17]. When the furnace reaches its target melt temperature of at least 650 degrees, the aluminium is introduced into the furnace and allowed to melt completely. Followed by stirring at 650 rpm for 20 minutes to create a vortex where the multiwalled carbon nanotubes are introduced using powder feeder[4], [13]. Once done, the melt undergoes ultrasonication for 15 minutes to facilitate dispersion[7], [8]. The preheated hydroxyapatite is fed through the powder feeder and stirred for 20 minutes and followed by 15-minute ultrasonication treatment and a final 20-minute stirring cycle is performed to ensure complete dispersion[8], [14], [15]. Then the molten composite is introduced to the preheated mould and pressure of 50MPa is instantly applied and maintained until solidification, after which the finished component is removed from the mould ready to be cut for required testing standards[9], [10], [13]. Figure 1 illustrates the schematic representation of the overall process for the fabrication procedure.

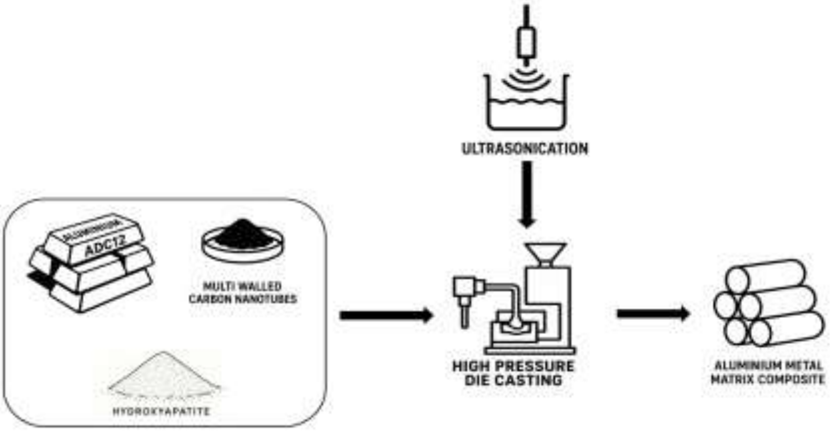

Fig. 1) Schematic representation of the fabrication process of aluminium metal matrix composite.

2.3 Process parameters

The process parameter optimization was conducted using Taguchi method for enhancing process efficiency by adapting L9 orthogonal array presented in Table 3, allowing efficient assessment of influence of several key process parameters, including weight percentage of the nanotubes, hydroxyapatite and furnace temperature for the fabrication of the metal matrix composite[9], [10], [17]. The process parameters maintained constant throughout all experimental trials are summarised in Table 1, while the variable parameters defined within the L9 orthogonal array are detailed in Table 2[17].

Table 1) Constant parameters

Parameter	**Constant value**
Stirring Speed	650 RPM
Ultrasonication time	2 parts each 15 minutes
Mold temperature	400
Reinforcement preheat temperature	350
Stirring time	4 parts each 20 minutes

Table 2) optimized parameters in accordance with L9 orthogonal array

Specimen ID	**Wt.% of multi-walled carbon nanotubes**	**Wt.% of Hydroxyapatite**	**Furnace Temperature (°C)**
312	0.75 %	0%	775°C
213	0.25 %	0%	825°C
221	0.25 %	0.25 %	725°C
232	0.25 %	0.75 %	775°C

The mass of the aluminium alloy required for casting was calculated from the mould volume and the alloy density, for minimising material usage during high pressure die casting.[9], [10]

The volume calculation of mould using density formula helps determining the mass of aluminium required

Aluminium ADC12 density : $\rho_{\mathrm{ADC12}} = 2.74\ \mathrm{g/cm^3}\#1$

Mould geometry : Diameter – 50 mm, Height – 200 mm.

The volume of the mould :

$$V = \pi r^2 h \qquad (1)$$

$$V = \pi\{2.5\ cm\}^2\{20\ cm\} \qquad (2)$$

$$V \approx 392.70\ \mathrm{cm}^3 \qquad (3)$$

Radius conversion :

$$r = \frac{50\ \mathrm{mm}}{2} = 25\ mm = 2.5\ cm \qquad (4)$$

Height conversion :

$$h = 200\ \mathrm{mm} = 20cm \tag{5}$$

Using the density formula :

$$m = \rho V \tag{6}$$

$$m = 2.74\ \mathrm{g/cm^3} 392.70\ \mathrm{cm^3} \tag{7}$$

$$m \approx 1075.998g \tag{8}$$

The precisely calculated mass of ADC12 aluminium alloy required for casting based on the mould geometry is 1.07 kg to be consistently used across all experimental trails.

Table 3) Orthogonal L9 design matrix

Factor 1	**Factor 2**	**Factor 3**
1	1	1
1	2	2
1	3	3
2	1	3
2	2	1
2	3	2
3	1	2
3	2	3
3	3	1

2.4 Experimental studies

2.4.1. Impact toughness evaluation (Charpy V-notch method)

The Charpy V-notch impact test employed to evaluate fracture resistance and its energy absorption capabilities under dynamic loading conditions[16]. This method uses a V-notched specimen that is simply supported at both ends and fractured by the impact of a swinging pendulum at the notch, concentrating stresses and promoting brittle fracture. The difference between the pendulum's initial potential energy and the residual energy after fracture is calculated to quantify the energy absorbed by the composite, expressed in joules[16]. The standard dimensions used for the Charpy V-notch test is illustrated in Figure 2.

Fig 2) Specimen dimensions for Charpy V-notch impact test

2.4.2. Microhardness evaluation (Vickers method)

This test is used to map detailed hardness across various microscale phases and grain boundaries to understand localized mechanical properties to target very small areas of grains[16]. The Vickers microhardness number (HV) was obtained from the applied load to the surface area of the indentation, reflecting the material's resistance to plastic deformation using diamond shaped indenter[16]. The values were measured by applying 100gram-force at dwell time of 10 seconds on a polished mirror finished surface.

2.4.3. Macrohardness evaluation (Rockwell method)

The Rockwell hardness test was employed to determine the hardness of metal accurately by applying minor preload to a hardened steel ball indenter for aluminium alloys to establish a zero-reference depth followed by a major load up to 150 kgf for fixed dwell on a 50mm diameter specimen[16]. The depth difference of indentation from the initial reference is measured and converted into Rockwell hardness number where higher numbers correspond to harder material[16].

3. Results and discussions

3.1. Impact toughness evaluation (Charpy V-notch method)

The impact energy values obtained from Charpy V-notch impact test are shown in the table 4 below.

Table 4) Impact energy values obtained from Charpy V-notch testing.

Sample ID	Wt. % of multi-walled carbon nanotubes	Peak impact energy (J)	Average impact energy (J)	Error (J)
232	0.25%	2.13	1.93	0.20
221	0.25%	1.35	1.22	0.13
213	0.25%	1.82	1.78	0.04
312	0.75%	1.73	1.43	0.30

Sample 232 shows a significantly higher impact energy of 2.13J than the others (as illustrated in Figure 3). indicating the improved ductility and ability to undergo plastic deformation before fracturing[6], while the lower values shown in sample 221 (1.73 J) indicates increased brittleness due to suboptimal processing temperatures (725 °C)[8]. The reinforcement composition directly influences energy absorption, as nanotubes can create barriers to crack proposition and improve ductility but due to melt temperature causing inhomogeneous dispersion can act as crack initiation sites[4], [6]. Well dispersed reinforcements impede initiation and crack propagation, leading to more energy absorption and tougher behaviour as shown is sample 213 with highest melt temperature of 825 °C[8]. A ceramic material contributes to strength but can introduce brittleness at high concentration due to its limited plastic deformation capacity[15], [16]. The sample with highest toughness, that is 232 indicates microstructural balance between multi-walled carbon nanotubes and hydroxyapatite that maximizes energy dissipation preventing excess brittleness[4], [6], [14]. The lower impact can be attributed to suboptimal reinforcement content leading to easier crack propagation and less energy absorption[6], [16].

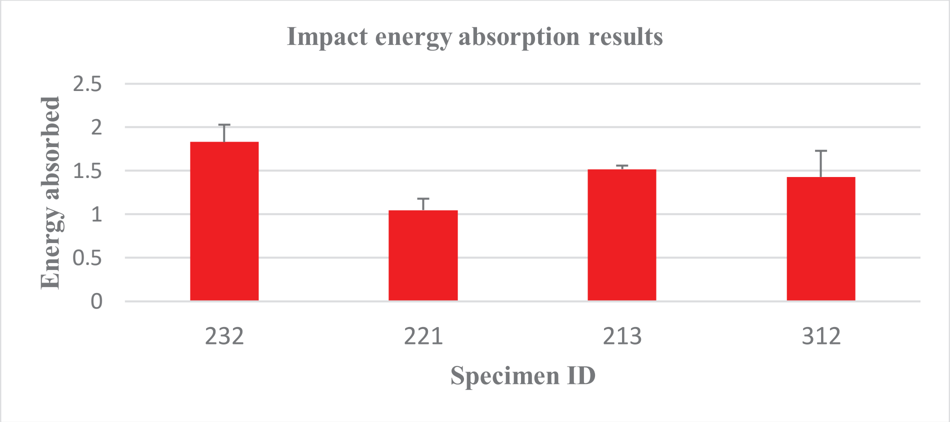

Fig 3) Charpy impact test results showing the energy absorption of the aluminium MMC

3.2. Macrohardness evaluation (Rockwell method)

Table 5) Indentation hardness results obtained using Rockwell testing.

Sample ID	Wt.% of multi-walled carbon nanotubes	Peak Hardness (HRB)	Average hardness (HRB)	Error (HRB)
213	0.25%	59.2	56.2	3.0
221	0.25%	49.2	46.6	2.6
312	0.75%	57.2	56.14	1.06
232	0.25%	52.4	47.66	4.74

Table 5 shows that the sample 312 and 213 exhibits the highest hardness of around 56 on HRB scale, while 221 and 232 shows a hardness of around 47 on HRB scale[16]. The specimen 312 with the highest multi-walled carbon nanotubes concentration shows high hardness[4], [5]. The elevated hardness values are attributed to the highest melt temperature employed during the casting of the specimen which can enhance wetting and promote uniform integration of reinforcements, resulting in homogenous reinforcement integration, increasing energy required for localised deformation resulting in elevated hardness[7], [8]. The samples subjected to lower melt temperature exhibit poor hardness values namely sample 221 and 232 which can be attributed to its poor reinforcement distribution and agglomeration restricting load transfer[6], [8]. The comparative Rockwell hardness analysis, as illustrated in Figure 4, indicates that the specimen 213 and 312 benefited from higher melt temperature and increased multiwalled carbon nanotubes incorporation, achieving superior resistance to indentation whereas the samples 221 and 232 were limited by its process parameters[4], [7], [16].

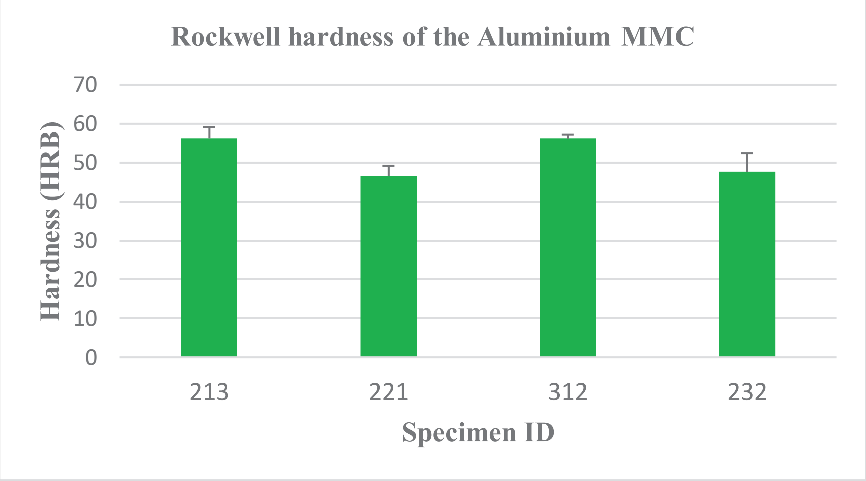

Fig 4) Rockwell hardness values of the aluminium metal matrix composite.

3.3. Microhardness evaluation (Vickers method)

Table 6) micro hardness values obtained from Vickers indentation

Specimen ID	Wt.% of multi-walled carbon nanotubes	Mean hardness (HV)	Peak hardness (HV)	Lowest hardness (HV)	Standard deviation (HV)
213	0.25%	122.9	157.5	90.3	19.5
221	0.25%	90.34	120.6	71.3	17.7
232	0.25%	107.4	124.7	96.7	10.6
312	0.75%	107.1	142.2	93.8	14.6

The hardness trends as summarized in Table 6 and further visualised in Figure 5 show that the specimen 213 has the highest and most consistent hardness values with highest of 157.5 HV like the Rockwell hardness values followed by sample 232, 312 and lowest hardness noted in 221[16]. The role of multi-walled carbon nanotubes in hardness is to enhance the aluminium matrix composites by impeding dislocation motion to facilitate increased load transfer[4], [5], [6]. The samples other than 312 consist of similar weight percentages of multi-walled carbon nanotubes, yet sample 213 shows the highest hardness, due to the elevated furnace melt temperature which provides better wetting and dispersion of multi-walled carbon nanotubes into the aluminium melt forming improved interfacial bonding which can correspond to better load transfer among the reinforcement and the metal matrix[7], [8]. The samples attributed to lower melt temperature (232 and 312) has similarly lower hardness values due to agglomeration from the lower melt temperatures[6], [8]. The 221 sample shows the lowest hardness which can corresponded to the lowest furnace temperature, which could cause poor dispersion and formation of increased agglomerates and porosity[6], [9], [10].

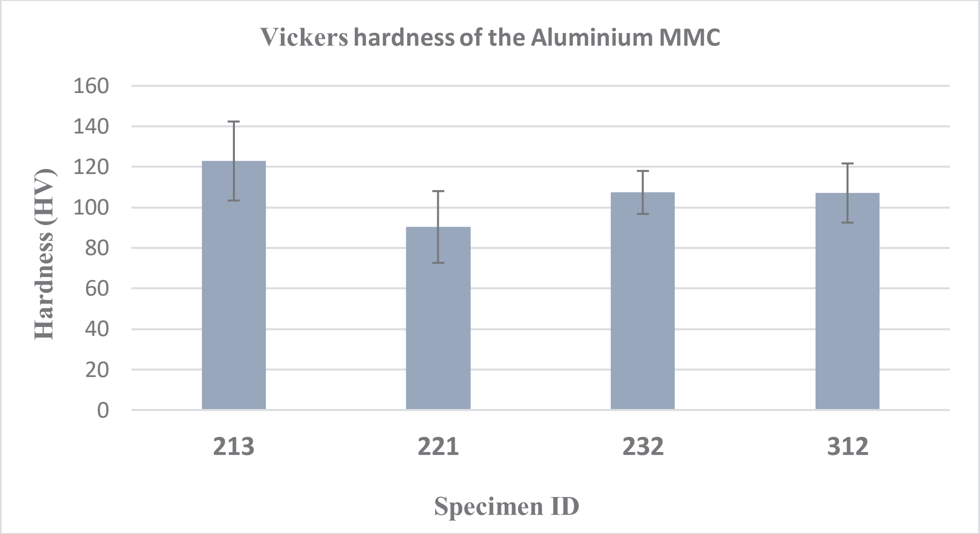

Fig 5) Vickers micro hardness values for the aluminium metal matrix composite.

4.Conclusion

This research evaluates the mechanical properties of ADC12 aluminium alloy metal matrix composites reinforced with multiwalled carbon nanotubes and hydroxyapatite under varied processing conditions. It is observed from the study that the processing parameters and reinforcement composition has a significant impact upon the mechanical characteristics of the aluminium metal matrix composites. The specimen processed at the highest melt temperature (specimen AL-213) exhibits higher hardness metrics indicates the impact of furnace temperature on matrix-reinforcement integrity as evidenced by the inconsistencies shown in specimen AL-221, which was fabricated under lower furnace temperature, which causes poor wettability and weak interfacial bonding between the reinforcement and aluminium matrix. Maximum energy dissipation found in specimen AL-232 withstanding the highest impact energy suggests an ideal proportion between reinforcement and furnace temperature. Specimens with optimal reinforcement content and furnace temperatures show consistent results in micro and macro hardness values highlights the importance of strategic optimisation of reinforcement composition and furnace melt temperature in improving the mechanical performance of ADC12 aluminium matrix. Integration of advanced analytical techniques and extensive characterization methods such as machine learning driven parameter optimisation can contribute to advancements in manufacturing practices. Further investigation would systematically explore higher reinforcement concentrations, quantify reinforcement agglomeration dynamics in combination with restructured process parameters to assess the full performance range of the composite. The present work proves a strong basis for developing advanced metal matrix composites suitable to effectively transition to industrial level production demands while retaining the performance observed in laboratory-scale investigations for demanding engineering applications.

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