

Synthesis and Characterization of Novel Nickel Coated Carbon Fibre Rod Reinforced Aluminium Metal Matrix Composite Material

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Abstract

A carbon fibre (CF) in the form of a rod (continuous fibre) was used as reinforcement due to its superior strength and modulus properties. Carbon fibre rod reinforced aluminium 6061 alloy metal matrix composite material was synthesized using the stir casting liquid metallurgy route. Aluminium based metal matrix composite materials are light in weight. Very few researchers were worked on carbon fibre rod reinforced metal matrix composites (MMC). Electroless nickel deposition on carbon fibre rods (2mm and 3mm diameter) were carried out for improvement in wettability. On carbon fibre rods, the effectiveness of electroless nickel coating was validated using SEM (scanning electron microscopy) and EDAX (Energy Dispersive X-Ray Spectroscopy) analysis. A further coating thickness of nickel was improved using nickel electroplating. In a cast iron mould, nickel-coated carbon fibre rods were arranged in a circular pattern. Completely degassed molten aluminium 6061 alloy was poured in cast iron mould at 600-700 °C. The enhancement in Tribological and mechanical properties is always a prerequisite for technological advancement in automobile sector. From synthesized composites (11.11% Vol. CF and 25% Vol. CF reinforcement) specimen were prepared for density tests, bulk and microhardness tests, friction and wear tests. Synthesized composite has low density, increased bulk and microhardness, low coefficient of friction and reduced wear rate in comparison with aluminium 6061 alloy.

Keywords: Carbon Fibre (CF) rod, MMC, Nickel coating, Casting, Mechanical, Tribological

1. Introduction

Carbon Fibres are useful as reinforcing elements in the diversity of matrix materials because of their high modulus, high strength, chemical reaction resistance including improved wear resistance and hardness[1–3]. Traditional metal alloys have specific characteristics that limit their usage in aeronautical and automotive applications. Traditional structural materials are less robust and heavier than carbon fibre reinforced composite materials. Due to their small weight and high strength properties, Aluminium alloys are employed in a variety of automobile applications. Poor hardness and limited wear resistance prevent them from being used in several applications. Composite materials are made up

of two basic components: reinforcement and matrix. The properties of composite materials are determined by the reinforcing and matrix materials used, as well as the processing method. The best features of two heterogeneous materials are combined in composite materials. Metal Matrix Composites (MMC) are recognized for having high specific strength, high stiffness, and better wear resistance due to improved hardness. Aluminium alloy was found useful as a matrix material for composites in the fabrication of various automobile components. Carbon fibre reinforced aluminium metal matrix composite material is prominent for automobile application due to its lightweight, high strength and remarkable wear properties [4–14].

In ceramic, metal, and polymer matrices, carbon fibre can be used as a reinforcing material because of its admirable properties. Carbon fibre reinforced polymer composites are extensively utilized in several applications; however, carbon fibre (CF) reinforced metal matrix composites have yet to reach that point due to processing difficulties. In the liquid metallurgy route squeeze casting, stir casting, and infiltration with squeeze casting is the most common methods for processing carbon fibre reinforced metal matrix composites. The stir casting liquid metallurgy route was economical and flexible for the synthesis of metal matrix composite material [15–18].

One of the most significant issues encountered in the synthesis of carbon fibre reinforced aluminium matrix composites is poor wettability. For this stir casting route, the aluminium alloy was used in the molten phase at 600-700 °C. To avoid the destruction of carbon fibres during processing, metallic (nickel or copper) coating on carbon fibres is used to improve interfacial characteristics. Nickel coating enables the wetting of molten aluminium with Carbon fibres. This metal coating improves chemical bonding between carbon fibre and metal matrix material [19–25]. Magnesium is a very effective surfactant, which results in improved wettability. During the synthesis of composite material, an optimal quantity of magnesium is required [19–21].

Carbon fibres in the form of short fibre or whisker fibres are used in foregoing MMC synthesis. These form of carbon fibres were aggregated during stir casting in molten metal which leads to property deterioration. The present work covers the synthesis of aluminium 6061 alloy and nickel coated carbon fibre rods composite material using the stir casting liquid metallurgy technique. The CF rods overcomes

the problem of aggregation and there is no need to manage all the parameters during the stir casting. Previously only electroless nickel or copper coating was used to deposit metal on CF rod surface. In this research work at first electroless nickel plating was performed which was followed by electroplating of nickel on CF rod surface. Commercially available EN solution was used for electroless nickel plating CF rods which reserves the time for coating. This dual plating results in nickel coating thickness improvement leads to negligible damage to CF rods and improvement in mechanical and tribological properties. The dual nickel plating was verified using SEM and EDAX analysis for coated and uncoated CF rod. Present work includes innovative use of CF rods as reinforcement in Al 6061 MMC and novel dual nickel plating. Fabricated composites were studied for their mechanical and tribological characteristics. Synthesized composite has low density, increased bulk and microhardness, low coefficient of friction and reduced wear rate in comparison with conventional aluminium 6061 alloy. The synthesized composites are a promising material for automobile brake drum and disc applications due to their outstanding wear resistance and low density.

2. Experimental Procedure

2.1. Material Selection

Aluminium 6061 alloy was considered a matrix material. Matrix material was used in the ingot form. The chemical composition of Aluminium 6061 alloy after casting is given in Table 1. Aluminium 6061 alloy was provided by Fen Fee Metallurgicals, Bangalore. PAN based carbon fibre in the rod form (2 mm and 3 mm diameter) was selected as a reinforcement material. Carbon fibre rods were provided by RC Dhamaka, Bangalore.

Table 1:

Chemical Composition of Aluminium 6061 Alloy

Element	Si	Mg	Fe	Mn	Cu	Zn	Cr	Pb	Ti	Al
Weight %	0.05	0.08	0.03	0.01	0.25	0.08	0.17	0.0032	0.019	Balance

2.2. Electroless Coating of Carbon Fibre Rod

Electroless nickel coating was performed to improve the wettability of carbon fibre rods in Aluminium 6061 alloy. Electroless nickel on carbon fibre includes cleaning, etching, sensitization, activation and electroless coating. Cleaning, etching, sensitization and activation, these steps were involved in pre-coating. Each step in pre-coating was followed by neutralization. Deionized water was used for neutralization after each step. The initial dust and finish layer was removed by using acetone (ultrasonic cleaning). Etching was done on the cleaned fibres using ammonium fluoride and sodium chloride. Etching was done for pore formation to increase surface area and establish hydrophilic spots that can absorb the etchant quickly. Etching follows sensitization using stannous chloride and activation using palladium chloride solution. During sensitization and activation, there

was the formation of surface micro-cavities for the autocatalytic electroless metal plating process to be started. Details regarding each step (ultrasonic cleaning, etching, sensitization, activation and neutralization) were mentioned in Table 2. For electroless nickel coating commercial EN832 solution provided by Marshal Laboratories, Pune was used. Pre-coated carbon fibre rods were coated using EN 832 solution at $83 \pm 2^\circ\text{C}$ as shown in Fig. 1. This solution includes nickel ions, stabilizer, reducing agent, and buffering agent. Effect of pH value on nickel coating was studied and complete electroless nickel coating was carried out at pH=5. Nickel electroplating was used to enhance the thickness of the nickel coating on carbon fibre rod [19-22].

Table 2:

Electroless coating of nickel on carbon fibre rod

Sr. No.	Operation	Chemical used	Composition	Duration (min)
1	Cleaning	Acetone	-	30
2	Cleaning	Deionized Water	-	5
3	Etching	$\text{NH}_4\text{F}/\text{NaCl}$	150g/l/150g/l	10
4	Neutralization	Deionized Water	-	5
5	Sensitization	SnCl_2/HCl	10g/l/40ml/l	240
6	Neutralization	Deionized Water	-	5
7	Activation	PdCl_2/HCl	0.5g/l/10ml/l	15
8	Neutralization	Deionized Water	-	5
9	Electroless Nickel Coating	EN832 solution	EN832 Solution	35



Fig. 1: Electroless Nickel Plating setup

Morphology of nickel coated and uncoated carbon fibre rods was studied by using EDAX (Energy Dispersive X-Ray Spectroscopy) analysis and SEM (Scanning Electron Microscopy). SEM analysis was carried out for the surface

and a cross-section of the carbon fibre rod. EDAX analysis was performed on the surface of coated and uncoated carbon fibre rod. SEM-EDAX analysis of Coated and uncoated CF rod samples were done using JSM-6390 (SEM) at Sophisticated Test and Instrumentation Centre, Cochin, Kerala.

2.3. Synthesis of Composite Material

For the synthesis of metal matrix composite material, aluminium 6061 alloy was used as a matrix material. Split type Die for casting was designed and fabricated. Cast iron was used as a die material for aluminium alloy casting. Considering specimen size required for various characterizations circular rod shape (bar) was finalized for casting. Dimensions of the circular rod were finalized.

Synthesis of Aluminium Metal matrix composite material (AMMCM) was done at coal based furnace. Weighted (5 kg) aluminium alloy ingots were placed in a furnace for preheating (around 400-450 °C). The adjacent furnace was started and heated. The temperature was maintained at 700-800 °C for aluminium alloy melting. Magnesium was added to the Aluminium 6061 alloy to improve wettability, and the molten alloy was stirred at 700–800°C at 200–250 rpm [15-18,27]. Preheating of cast iron die was done at temperature 200-250 °C for preparation of bars. Using c-clamp and the green sand cast iron die was kept ready for pouring molten aluminium alloy. Degassing of molten aluminium alloy was carried out using a degassing tablet. Completely degassed molten aluminium alloy was poured in casting die and after solidification aluminium alloy rods were separated [15-16].

Carbon fibre rods were reinforced in percentage volume fraction in the aluminium matrix material. Nickel Coated Carbon fibre rods were mounted in the casting die before molten aluminium alloy pouring. To maintain the upright posture of the carbon fibre rods, a circular plate with holes (having the same diameter as the carbon fibre rod) was kept below the casting die. Five carbon fibre rods were arranged in such a way that one was at the centre and the remaining four in a circular array around centre one. The diameter of the cast AMMCM bar was 35 mm. In preheated casting die (in which coated carbon fibre rods were placed), a degassed molten aluminium alloy was poured. After solidification, AMMCM cast rods were collected [15-18].

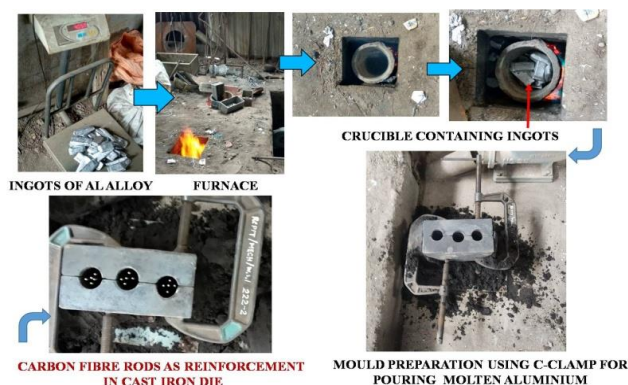


Fig. 2: Synthesis of Aluminium Metal Matrix Composite Material

2.4. Characterization of Composite Material

Specimen required for characterization was prepared using laser machining. The Archimedes concept was used to determine the density of carbon fibre rod reinforced aluminium metal matrix composites material. Specimens were prepared for tribological and mechanical characterization from cast rods.

For Brinell hardness (BHN) means bulk hardness test ASTM E10 standard was used. ASTM E92-17 standard was used to perform Vickers hardness (VHN) means microhardness test. A hardness test was performed at Mattest Laboratory, Aurangabad (NABL Accredited). A cylindrical disc was used as a specimen for hardness testing. The cylindrical disc has a 30 mm diameter and 10 mm thickness. A hardened steel ball indenter of diameter 2.55 mm was used for the bulk hardness test. A load of 187.5kgf was applied for 15 seconds. An average of 6 different readings was considered for the final BHN value. The diamond indenter was used for the microhardness test. For microhardness, the test was performed in the vicinity of the CF rod and away from it. Six different measurements were taken and the final VHN value was the average of them. A bulk hardness and microhardness test was carried out for cast aluminium 6061 alloy and its composites.

ASTM G99-95 standard was used to prepare specimens of the wear test. A tribological test was performed on a pin on disc tribometer (Make: Ducom Instrument Pvt. Ltd. Bangalore) at a research laboratory, PREC, Loni, Pune University. For tribological characterization, the cylindrical pin was prepared for both cast aluminium alloy and cast AMMCM. A cylindrical pin of diameter 6 mm and length 25 mm was used as a wear (tribological) pin. The test specimens end surfaces were polished and flattened. The cast iron disc was used as a counter disc which has a hardness of 60 HRC and surface finish was 1 µm. The time allotted for the test was 30 minutes. The sliding velocity and load applied had been used to compute the wear rate. From the investigation of various research papers, it was observed that as the sliding velocity and load applied increases wear rate also increases [6-8]. After a detailed investigation of research papers, 20 N Load and 0.51 m/s sliding velocity was finalized for the tribological test. Wear loss was measured in the steady-state region. The coefficient of friction was evaluated considering frictional and normal load data. Using height loss data, the wear rates were evaluated in terms of volumetric wear loss per unit sliding distance. Friction and wear test was performed for cast aluminium 6061 alloy and its composites.

Comparison of results (Tribological and Mechanical characterization) for cast aluminium 6061 alloy and its composites will be mentioned in the Result and Discussion.

3. Result and Discussion

3.1. Characterization of Nickel Coated and uncoated Carbon Fibre Rod

To improve the chemical bond between carbon fibre (CF) rod reinforcement and aluminium 6061 alloy matrix, successful nickel coating on CF rod was necessary. The hardness and wear properties of the synthesized composite were significantly enhanced by the improved bonding between the matrix and the reinforcing material. Electroless nickel coating on the CF rod was carried out. Coating morphology on CF rod surface was studied using SEM-EDAX analysis. For coated

and uncoated CF rods, SEM analysis was performed at its surface and cross-section to observe nickel coating at high magnification. Elemental composition at the coated and uncoated surface of the CF rod was observed using EDAX analysis [19-22].

3.1.1. SEM-EDAX Analysis of uncoated Carbon Fibre Rod

Specimens for nickel-coated and uncoated carbon fibre rods were prepared. SEM analysis was used to investigate the surface of nickel coated and uncoated carbon fibre rods. Fig. 1 shows SEM analysis of Nickel Coated and uncoated CF rod surface. The surface was observed at 50 micron magnification. It was observed that coating of nickel was done uniformly over the carbon fibre rod surface.

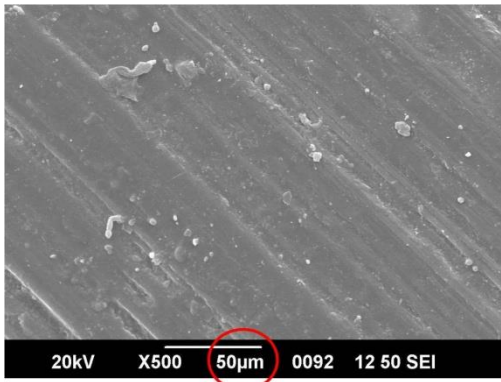


Fig. 1: SEM of uncoated CF rod surface

SEM analysis was also carried out for the carbon fibre rod cross-section. Fig. 2 shows SEM analysis of Nickel Coated and uncoated CF rod cross-section. The cross-section of the carbon fibre rod was completely coated with nickel, which was observed at 500 micron magnification. From complete SEM analysis for cross-section and surface of carbon fibre rod, it was concluded that nickel coating was uniformly distributed.



Fig. 2: SEM of Nickel Coated and uncoated CF rod cross-section

Fig. 3 shows the EDAX spectrum of the uncoated CF rod surface. It was found that carbon was present at the surface of the CF rod. Important EDAX analysis was carried out for nickel coated CF rod surface. Fig. 4 shows the EDAX spectrum of Nickel Coated CF rod surface. It was clearly observed that nickel was coated over the CF rod surface. Nickel compound was found over carbon (CF rod surface) during the Elemental composition study. No void was present on the CF rod surface which results in uniform nickel coating. After electroless nickel coating, nickel electroplating was

done to increase the thickness of the coating. This effective nickel coating results in a good bond between CF rod reinforcement and aluminium 6061 alloy matrix material.

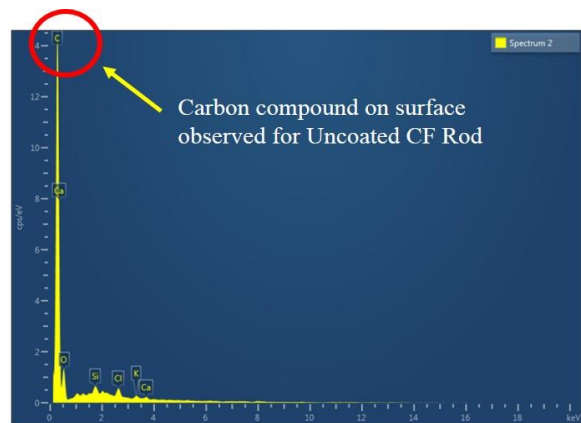


Fig. 3: EDAX spectrum of uncoated CF rod surface

Table 3:

Compositional Analysis of uncoated CF rod surface

Sr. No.	Element	Weight (%)	Atomic (%)
1	C	83.98	87.93
2	O	14.76	11.6
3	Si	0.35	0.15
4	Cl	0.51	0.18
5	K	0.21	0.07
6	Ca	0.19	0.06

3.1.2. SEM-EDAX Analysis of Nickel Coated Carbon Fibre Rod

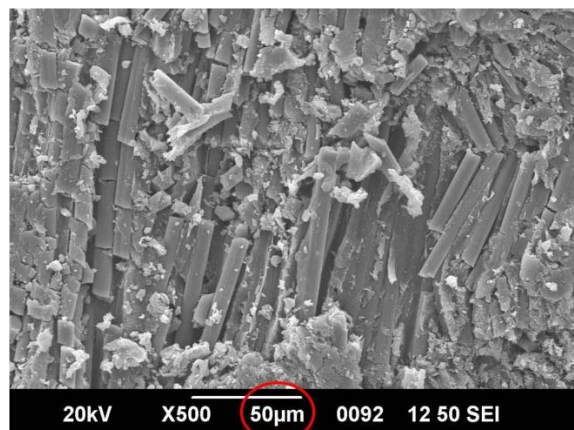


Fig. 1: SEM of Nickel Coated CF rod surface



Fig. 2: SEM of Nickel Coated and uncoated CF rod cross-section

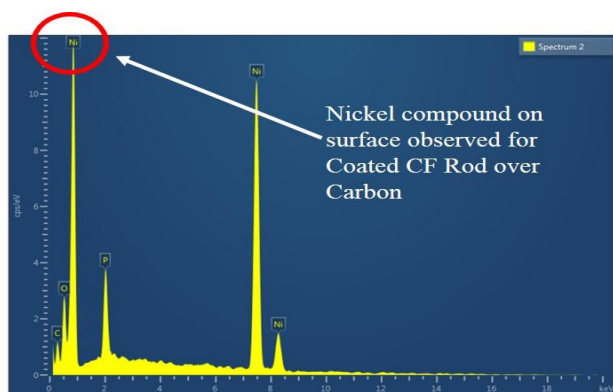


Fig. 4: EDAX spectrum of Nickel Coated CF rod surface

Table 4:

Compositional Analysis of Nickel Coated CF rod surface

Sr. No.	Element	Weight (%)	Atomic (%)
1	C	15.92	40.95
2	O	8.8	16.98
3	P	5.24	5.23
4	Ni	70.04	36.84

3.2. The density of Composite Material

Using Archimedes concept, the density of Aluminium 6061 alloy and carbon fibre (CF) reinforced composite materials were calculated. The density of Aluminium 6061 alloy and CF reinforced composite materials were shown in Fig. 5. As the percentage volume fraction of carbon fibre rod reinforcement in Aluminium alloy increases, then there is a reduction in density. This reduction in density is due to the low density of carbon fibre rods as compared to Aluminium alloy matrix material. It is observed that, the density of Al6061+11.11% Vol. CF reinforced and Al6061+25% Vol. CF reinforced composite material is 8.45% and 13.28% lower than aluminium 6061 alloy respectively [6-9].

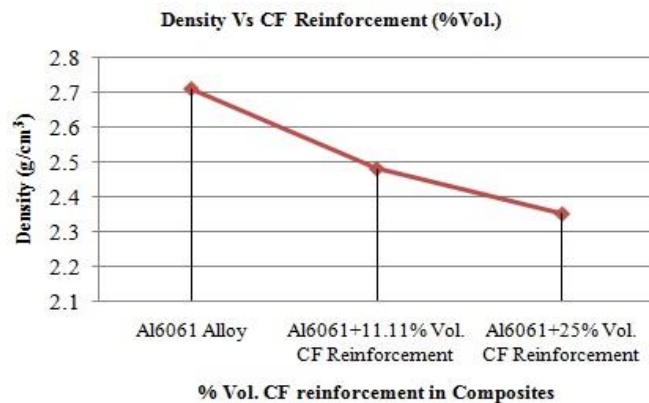


Fig. 5: Density of Aluminium 6061 alloy and CF reinforced composites

3.3. Mechanical Characterization

Fig. 6 shows bonding between CF rod and Al 6061 alloy of metal matrix composite material. At micron level (SEM) great bonding between CF rod and Al 6061 alloy due to the nickel coating was observed. The actual image shows no pull out of CF rods from the cast composite bar. Because of the nickel coating on carbon fibre, there is no chemical interaction between carbon fibre rods and the aluminium alloy matrix, which avoids carbon fibre deterioration [7-9].

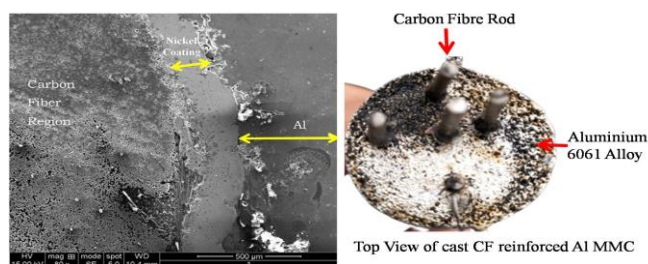


Fig. 6: SEM and actual image of bonding between CF rod and Al 6061 of composites.

One of the most important aspects that impact the wear rate of composite material is the surface hardness. Brinell hardness (BHN) means bulk hardness was measured for Aluminium 6061 alloy and CF reinforced composite materials as shown in Fig. 7. The bulk and microhardness of the composite material improve as a result of the strong bonding between reinforcement and matrix material.

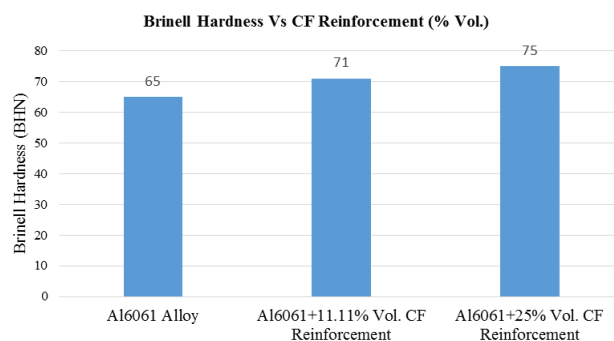


Fig. 7: Brinell hardness (BHN) of Aluminium 6061 alloy and CF reinforced composites

As the percentage volume fraction of carbon fibre rod reinforcement in Aluminium alloy increases, then there is an increase in bulk hardness (BHN). It is observed that, the bulk hardness (BHN) of Al6061+11.11% Vol. CF reinforced and

Al6061+25% Vol. CF reinforced composite material is 9.23% and 15.38% more than aluminium 6061 alloy respectively.

Measurement of Vickers hardness (VHN) means microhardness was done for Aluminium 6061 alloy and CF reinforced composite materials as shown in Fig. 8. Microhardness measurement was carried out in the vicinity and away from the CF rod. As the percentage volume fraction of CF reinforcement increases in matrix material, there is an increase in microhardness. Nickel is also diffused in the vicinity of CF rod during synthesis of composite material which also results in increased microhardness. The microhardness (VHN) of Al6061+11.11% Vol. CF reinforced and Al6061+25% Vol. CF reinforced composite material was increased as compared to aluminium 6061 alloy.

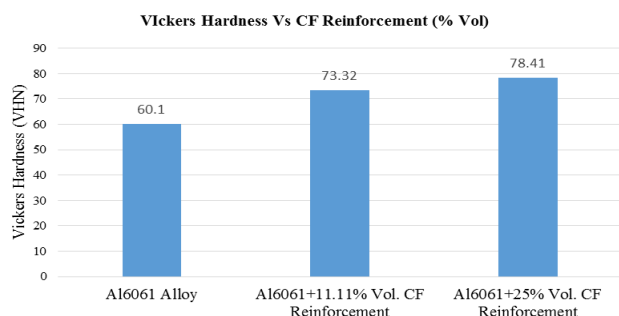


Fig. 8: Vickers hardness (VHN) of Aluminium 6061 alloy and CF reinforced composites

3.4. Tribological Characterization

As the percentage volume fraction of CF reinforcement increases, bulk and microhardness increase which results in superior wear resistance of composite material. Wear resistance was improved due to the great bonding between nickel coated CF rod and aluminium 6061 alloy, good lubricity between carbon fibres within the CF rod and no pull out of carbon fibres during wear test.

The coefficient of friction for Aluminium 6061 alloy, Al6061+11.11% Vol. CF reinforced and Al6061+25% Vol. CF reinforced composite material was observed in Fig. 9. It was concluded from tribological characterization that synthesized composite material shows a reduced coefficient of friction in comparison with aluminium 6061 alloys. It was found that the coefficient of friction of Al6061+11.11% Vol. CF reinforced and Al6061+25% Vol. CF reinforced composite material is 4.59% and 11.6% lower than aluminium 6061 alloy respectively.

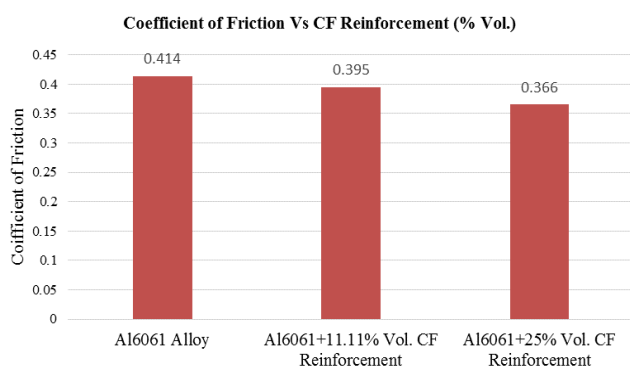


Fig. 9: Coefficient of friction for Aluminium 6061 alloy and CF reinforced composites

The sliding velocity and load applied had been used to compute the wear rate. From the investigation of various research papers [6-8, 13, 14], it has been discovered that as the applied force and sliding velocity rise, so does the wear rate. After a detailed survey, it was concluded that measurement of wear rate was done at 20 N applied load and 0.51 m/s sliding velocity using DUCOM pin on disc tribometer. Wear rate for Aluminium 6061 alloy, Al6061+11.11% Vol. CF reinforced and Al6061+25% Vol. CF reinforced composite material was as shown in Fig. 10.

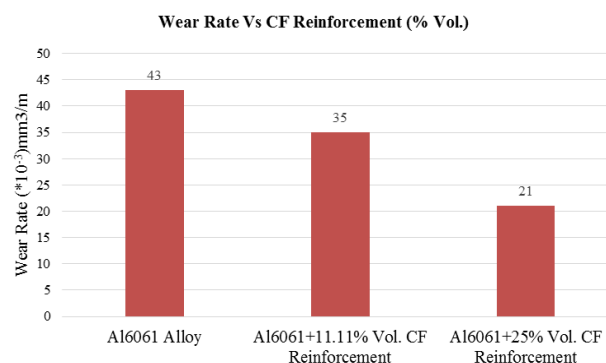


Fig. 10: Wear Rate for Aluminium 6061 alloy and CF reinforced composites

It was found that, increase in percentage volume CF reinforcement (up to 25%) in matrix material results in a reduction in wear rate. During the wear test, graphite was discovered between sliding surfaces, which functions as a solid lubricant and results in a decreased wear rate. Furthermore, it was found that the wear rate of Al6061+11.11% Vol. CF reinforced and Al6061+25% Vol. CF reinforced composite material is 18.6% and 51.16% lower than aluminium 6061 alloy respectively.

4. Conclusion

Important conclusions from the work are as below:

1. The stir casting liquid metallurgy route was economical and effective for the synthesis of Carbon fibre rod reinforced aluminium 6061 alloy metal matrix composite material.
2. Electroless coating methodology gives a uniform and effective deposition of nickel on carbon fibre rod surface observed using SEM-EDAX analysis. Improvement in a chemical bond between reinforcement and matrix material due to effective nickel coating on carbon fibre rod.
3. Remarkable 13.28 % reduction in density of synthesized composite material as compared to cast aluminium 6061 alloys.
4. Significant increase in bulk and microhardness of synthesized composites material as compared to cast aluminium 6061 alloy due to increase in CF reinforcement.
5. As compared to cast aluminium 6061 alloy, the synthesized composite material has a lower coefficient of friction (11.6%) and a lower wear rate (51.16%) due to superior bonding between reinforcement and matrix material.
6. Novel material has prospective application in automotive field for brake disc, brake drum etc. due to its enhanced tribological and mechanical properties.

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