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Growth of Ni–Cu–Zn electrolyte coated thin layer carbon fiber reinforced aluminium composite

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Abstract

This work presents the feasibility study of Nickel-Copper-Zinc coated carbon fiber reinforced Al6061 composite with emphasis on avoiding oxidation of carbon fiber at relatively higher temperatures. The mechanical properties, microstructure and damping analysis of a novel Ni–Cu–Zn coated Al6061 composite were investigated. The tensile strength of coated composite and hardness gradually increases due to grain refinement of carbon fibers formation. The elongation of Al6061 alloy and Ni–Cu–Zn coated Al6061 composites are 7.4% and 6.9% respectively. Microstructure study reveals low wettability of the carbon fiber with the matrix resulting in the carbon fibre reinforcements getting separated with the aluminium matrix during loading.

Introduction

Polymer composites with nano carbon fibers serve a broad range of applications. Polymer composites are excellent friction and wear performance after being modified with functional fillers and reinforcements. The elastic modulus and tensile strength of carbon nanotubes to enable increased load transfer and improved mechanical strength of polymer nano composites [1]. Xu et al reported that increase in elastic modulus of silicon in polymers by damping capacity by 673% and toughness by 39% [2]. Like wise Synthetic fibers such as carbon, glass, and kevlar used in polymer composites for aerospace application are replacing the secondary structure with fiber–polymer composites [3]. Carbon fiber reinforced aluminium (CFRA) composites having the major advantage of improved strength to weight ratio make them highly suitable for use in aerospace and also satellite applications [4]. Carbon fibre reinforced aluminium would be able to operate in severe environmental conditions unsuitable to traditional polymer matrix composites, owing to the high melt temperature and high environmental resistance of the carbon fibre aluminium composite [5]. Since the mechanical properties of the composite is governed by the rule of mixture depending on the extent of carbon fibre content in the aluminium matrix, it becomes extremely important to conduct studies, parametric in nature [6, 7]. When combined with a plastic resin and wound or moulded it forms carbon fiber reinforced plastic (CFRP) which has a very high strength-to-weight ratio, and is extremely rigid although somewhat brittle [8]. Rams et al [9] had investigated electro-less nickel coated short carbon fibres in aluminium matrix composites. Thus coated carbon fibres (CF) had been used for reinforcement with aluminium matrix composites by liquid processing. Electro-less nickel coating of the carbon fibres enhances their wettability and allowing the homogeneous distribution of the fibres in the matrix with reducing the micro segregation surrounding the fibres. Sanchez et al [10] were study on fabrication of aluminium composites reinforced with carbon fibres by a combination of centrifugal and investment casting. Some samples were coated with nickel to favour the reinforcement wetting by the molten aluminium alloy. Carbon fibres of volume fraction more than 7% was used and porosity values lower than 0.5% volume were obtained with this technique. Zhang et al [11] had presented work on low-cost continuous production of carbon fibre reinforced aluminium composites. In this study fabrication is usually conducted through pressure-assisted casting, or with the use of ultrasound to induce cavitations and encourage infiltration. The composites produced with ultrasound were well-infiltrated, and were tested in tension to determine their mechanical properties. Author concluded that the research was designed to determine a suitable low-cost
coating for carbon fibers that would protect the fibers from interfacial reaction with aluminum and improve its wetting characteristics.

Ramesh et al\cite{12} had investigated the compatibility of pitch based with aluminium matrix composites. Pitch based carbon fibres are derived from coal based pitches and petroleum based pitches. Aluminium is coated to pitch based carbon fibres using chemical vapor deposition under reduced pressure before fabrication and care had to be taken that without any degradation of carbon fibres. Thus coated fibres were heated at 773–873 K in air at 95.8 kPa and subjected for testing. The tensile strength of aluminium coated pitch based carbon fibres decreases as a function of Young’s modulus. Urena et al\cite{13} had investigated the interface and thermal expansion of carbon fibre reinforced aluminium matrix composites. A 55% volume fraction of carbon fibre was reinforced with aluminium 6061 were made-up by squeeze casting. Aluminium composite density was decreased to 2.64 g/cc with increase in tensile strength of 314 MPa compared with aluminium alloy. The Co-efficient of Thermal Expansion (CTE) of composites were decreased, which were much lower than that of the aluminium alloy this is due to the mechanical restraint imposed by fibres on the thermal expansion of the aluminium alloy. Author was concluded that the combination between aluminium alloy and fibres was well in composites and CTEs of composite varied between the heating temperatures, which were in a good state according to Schapery model.

Shirin et al\cite{14} studied that wear of aluminium matrix composites reinforced with 1% to 10% volume fraction of Al2O3 nano particles. Thus produced composites were tested; results indicated that the coated composite had higher resistance than the unreinforced alloy. Popovska et al\cite{15} had investigated mechanical and tribological properties such as Hardness, bending strength, wear and friction co-efficient of carbon fibre reinforced copper alloy composites prepared by the powder metallurgy technique. The maximum hardness occurs for 15% volume fraction composite while maximum bending strength occurs for 9% volume fraction carbon fibres. Rajkumar et al\cite{16} had investigated the Transition Electron Microscope (TEM) study of carbon fibre reinforced aluminium matrix composites influencing on mechanical properties and brittle phases. From literature study only few researchers have been used triple coated carbon fibre composites. The main aim of this study is to realize the CFRAl composite without any degradation in the strength of carbon fibre due to oxidation at relatively higher temperatures. This work also includes optimization of coating techniques (possible combination of different coatings) to prevent carbon fibre oxidation at higher temperatures for spacecraft applications.

Materials and methods

Al6061 is used as matrix material; it is a versatile heat treatable extruded alloy with medium to high strength capabilities. It contains magnesium (0.8% to 1.2%) and silicon (0.4% to 0.8%) as its major alloying elements\cite{17}. The reinforcement used for the composite is carbon fibre. Carbon fiber is a light weight material, which possesses very high modulus and strength, which is currently being tried out as reinforcement in light metals alloy. Carbon fibers in tow form having 3000 filaments and each filament of 6–7 μm diameter was obtained from Marktech Ltd, Bangalore, India. A sample of 300 mm length 3 K carbon fiber is subjected to testing in an electric furnace. Carbon fibre is placed in an electric furnace and fiber is heated up to 250 °C for about 10 min in air is shown in figure 1. Carbon fiber is taken out from the furnace and then it was observed that fibre gets oxidized as shown in figure 2. As such, carbon fibre requires coating before getting reinforced with the aluminium in order to avoid oxidation\cite{18}.

After coating the carbon fibers were subjected to heating in an electric furnace in order to check the oxidation of carbon fibers. Before casting carbon fibre is completely coated with nickel by electro-less and electrolyte plating to avoid the oxidation during the process\cite{19}. Carbon fibers was subjected to electro-less nickel coating, electro-less copper coating, finally subjected to electrolyte copper plating and electrolyte zinc plating followed by die casting for producing different specimen. Molten aluminium alloy were poured between the layers of the carbon fibres placed in the metallic mould. The developed carbon fibre reinforced composite specimen will be subjected to evaluating the density, the tensile strength, the stiffness and the damping improvements as a function of different coating and related parameters of carbon fibres.

**Metallic coating on carbon fibre composite**

Three coating techniques were tried and tested for the reduction in oxidation levels and increase in density levels. The three coating techniques are described in below.
Electro-less nickel coating
Electro-less nickel bath using hydro-phosphite as a reducing agent was used in the present study to deposit a thin adherent nickel-phosphite coating on carbon fiber. The details of the deposition composition used for electro-less nickel are given in table 1.

Ten carbon fibers groupings, each having length of 350 mm and overall weight 1.24 gms was taken in a beaker and soak cleaned by immersing in toluene and propane followed by acetone wash [20]. This was followed by distilled water rinse. This procedure was used to remove dust, oil and other unwanted materials from the surface of carbon fibers.

Electrolyte copper plating
Electrolyte copper plating is a process in which a layer of copper is deposited, in this case, on the electroless nickel and copper coated carbon fibers [21]. Electrolyte copper solution uses copper sulphate as the electrolyte, copper
as the anode and the metal on non metal to be plated as cathode. Electrolyte copper bath using copper sulphate as the electrolyte was used in the present study to deposite copper coating on carbon fibers. The details of the bath composition used for electrolyte copper deposition are given in table 2.

Before carbon fibers are immersed into the electrolyte solution one end of the fiber is tied to a hanging support and the other end is tied to some weight. This procedure is followed inorder to create electric conductivity in the carbon fibers subjected to copper coating. Each carbon fiber is completely dipped into the solution and allowed to coat for about 30 min. The current density used in the solution is 2 V and 50 amps.

Electrolyte zinc plating
After Ni–Cu coating process, the carbon fibers are subjected to zinc electroplating. Here, Zinc electroplating occurs in an electrolytic solution tank. At the beginning, the cell consists of an electrolyte, which is a substance containing free ions which are the carriers of electric current. Next an anode is connected to the positive side of the power source. It attracts negatively charged ions or anions. Cathode is connected to the negative side of the power source. This provides electrons to the cations to convert them back to the parent metal, a process called reduction. The piece is providing the zinc metal is the anode and the part is deposited on the metal surface is the cathode. Carbon fibres before subjected to electroplating surface pre-treatment is did by using acetone to remove the surface impurities after this rinse it in water and treated with solution of 20% HCL and 80% water. Then fibres are dipped in an electrolyte solution for 15 min and solution is maintained with a current density of 7 V and 50 amps. The details of the bath composition used for electrolyte zinc deposition are given in table 3. Zinc coated carbon fibre is subjected to heating in an electric furnace at 630 °C about 5 min in order to observe oxidation of carbon fibre. After heating carbon fibre, the strength was checked by hand pullout test. It is noted that coated fibre is not undergone complete degradation as in the case of uncoated carbon fibre.

Figure 3 shows the electric resistance furnace setup used for the melting aluminium material. The cleaned metal ingots are heated to a temperature of 750 °C–800 °C by placing in a graphite crucible at the temperature of 800 °C and the melting point of Al6061 is around 700 °C. A filament winding type of induction furnace is used. Later on scumming powder is added into the molten metal to float the impurities on the top surface of molten metal. Then a degassing agent in the form of Hexamethyelene diamine is added during the melting period. Likewise, Magnesium is added in small quantities to improve the wettability of the reinforcement particles with the base matrix. Pretreated (coated) Carbon fibres of quantity 5 and 10 numbers of strands are bunched by using a copper wire of 0.8 mm diameter and then placed vertically in the mold cavity. The cleaned metal molds are then prepared by bolting together each part tightly in order to hold carbon fibres vertically and also to minimize the leakage of aluminium. Then the mold is placed inside square molds filled with sand to hold the
carbon fibres inserted mold tightly. The pouring temperature was maintained at 750 °C. The melt was then allowed to solidify in the molds.

After solidification of molten metal in the mold, the same is opened to observe if molten metal has filled mold properly in the cavities of mold. The improper metal flow occurs, if any, due to copper wire used to tie bunch of carbon fibres and mold is not preheated therefore solidification is faster. Therefore further casting would be tried incorporating some changes in methodology, which is discussed further. A mild steel plate of 2 mm thickness with other specific dimension not very crucial is used as a base plate for casting.

A mild steel plate is drilled 3 holes of diameter 4 mm at different locations and zinc coated 5 numbers carbon fibres are inserted into one of the hole to make the fibres stand vertically. At the bottom of the plate carbon fibres are tied to the mild steel plate using copper wire. Mold is placed on the steel plate and is surrounded by sand in order to avoid molten metal leakage. The molten Al is then poured into the metal mold and the pouring temperature was maintained at 750 °C. After solidification the mold setup used to develop zinc coated composite is shown in figure 4.

Experimental details

The specimens are prepared for mechanical and microstructure study is presented in this section. The specimen prepared for density test is shaped by cutting the obtained cast in power axle using milling machine. The specimen dimensions are 45 mm length, 11 mm width and 8 mm thickness. Density of the composite sample is determined by Archimedes principle i.e., the volume of displaced fluid is equal to the volume of the object [14]. Initially the weight of the sample is noted using electronic weigh balance then water is taken in a dried 50cc measuring jar up to 40cc completely. Then the sample is immersed into the measuring jar completely, the volume of water displaced is determined and the density is calculated using standard formula.

Hardness test is performed using Wolpert Wilson, 402 MVD with a load of 100 g for duration of 15 s. The test is carried out at eight different locations on two opposite surfaces of both composite samples in order to contradict the possible effect of diamond indenter resting on the harder particles. The averages of all the eight readings were taken as hardness of the sample [13, 17]. After the hardness test, the specimen was cut into a cross section and used for microstructure study.

Tensile test is carried out at a strain rates of $1.2 \times 10^{-2}$ s$^{-1}$ using universal tensile machine according to ASTM E8 standard are shown in figure 5. From the results the consequent stress–strain curves is plotted to find data such as elongation and tensile strength coated and uncoated samples [13]. For microstructure study a small piece of the specimen 100 mm overall length, 6.2 mm gauge width, 26 mm gauge length and 8 mm thickness is selected using cutting tools [23] and the specimen was mounted in thermoplastic resin such that the specimen surface is exposed. Emery papers of different grades (200, 400, 600, 800, and 1000) are used for this test. The specimen used for microscopic examination is subjected for SEM photography [21].
The grain size of the two specimens is analyzed using Nikon Microscope LV150 with Clemex Image Analyser according to ASTM E112-96 standard. For damping test the specimens were shaped by the obtained cast in power axle and using milling machine. The specimen dimensions are 100 mm length, 10 mm width and 8 mm thickness. The prepared specimen is subjected to damping test as shown in figure 6. One end of the specimen is fixed horizontally to the bench wise tightly so that all degrees of freedom are constrained at the fixed end. High sensitive eddy current probe is placed under the free end of the test specimen in order to obtain decay curves. The force is applied at the free end of the specimen by striking on the top surface. Deflections are sensed by eddy current probe and decay curves of amplitude versus time are generated by programmed software connected to the eddy current probe was displayed on the screen.

Results and discussion

Hardness

The variation in Vickers hardness behaviour of Al6061 and Ni-Cu-Zn coated Al6061 composites are reported in figure 7. Hardness test was carried out at eight different locations on two opposite surfaces of both coated and uncoated samples in order to contradict the possible effect of diamond indenter resting on the harder particles. The average of all the eight readings were taken from the hardness sample is shown in figure 7. It is observed
from the graph that there is an increase in hardness of aluminium-carbon fibre composite as compared to aluminium 6061. This is due to the quick cooling rate of molten metal and large amount of copper zinc coated carbon fibres are reinforced with molten aluminium. However there is an increase in strength of aluminium composite from interface of carbon-fibre towards the matrix phase than uncoated material [26]. In addition the coated sample shows better control of nano particles of Ni–Cu–Zn with uncoated composites. This can be justified that the distribution of reinforcement particles reduces the casting defects [13].

**Tensile strength**
The tensile strength results of Al6061 and Ni–Cu–Zn coated Al6061 composites are discussed in this section. The cross sectional view of fractured specimens after tensile strength is shown in figure 8. The variation obtained from engineering stress-strain graph at the strain rates of $1.2 \times 10^{-2} \text{ s}^{-1}$ for Al6061 and coated carbon fibre composite is plotted as shown in figure 9. The tensile strength for carbon fibre reinforced Al6061 composite is obtained 139 MPa for an applied peak load of 8 KN and the tensile strength of Al6061 specimen is 121 MPa for an applied peak load of 6 KN. The tensile failure of CFRA specimen in operation as compared to aluminium
6061 specimen is because of the poor wettability of CF into the matrix resulting in the reinforcement missing the load path.

From figure 9 the curve establishes that failure strength also increases with volume content of fibres from inner to outer region [13]. The density of the fibre content increment also stabilizes the matrix of the composite ranges from 20% to 50%. As predicted from the curves the linear content of carbon fibre increases the sensitivity of strain rate which decreases the hardening coefficient of the carbon fibre composite [27].

The area of fractured cross section of material for coated composite is very low compared to the Al6061 is shown in the figure 8. CFRA composite specimen failure may also occurs because carbon fibres are dominate than the aluminium matrix as shown in figure 9, nearly 55%-60% of fibres are reinforced. However, tensile strength of composite gradually increases and depends on the ratio of carbon fibres reinforced to metal matrix [17].

**Total elongation**

The total elongation of cast Al6061 alloy and Ni–Cu–Zn coated Al6061 composites presented the corresponding elongation values are 7.4% and 6.9% respectively. It is clearly predicted that upon forging there is significant increase in ductility when compared with uncoated AA6061 [24]. This can be mainly due to formation and
dislocation of intermetallic and grain refinement in matrix material during coating of fabricated composites [17]. Author Pourhoosseini et al clearly mentioned that coating composites may decrease the elongation properties because of dislocation motion of carbon fibres [14].

Microstructure analysis
Figures 10 (a)–(d) shows the microstructure of Al6061 and Ni–Cu–Zn coated composite at different magnifications. Figure 10 (a) shows the micrographic image of pure Al6061 specimen. Figure 10(b) shows the micrographic image of Ni–Cu–Zn coated Al6061 specimen. It is observed that the extent of bonding between carbon fibre and copper interface is good. It also indicates that there is a good wettability between copper and aluminium interface [21]. Thus it is possible to reinforce carbon fibre in aluminium matrix by coating copper on carbon fibre and zinc becomes alloy with aluminium [20].

At cross sections of composite the coated layer clearly shows dislocation of fibre content. The chemical coated layers are exactly marked the images. A major aspect in the images of figures 10 (c), (d) presents many fibre layers and coating orientations are present in the structure region which increases the interfaces between the layers [23, 28].

Statistical grain size distribution is shown in the figures 11(a)–(c) according to ASTM E112-96 standard. The morphology image of the coated composite is shown in figure 11(a). The grain size distribution of Ni–Cu–Zn coated composite is shown in figure 11(b). It is observed that the improved grain size of the specimen increases the intercept of the grain structure [22].

The maximum grain size number is 9.28 and minimum grain size number is 3.21. It is observed from the histogram that the average grain size number 5.5 obtained from test result as shown in the histogram figure 11(c). Author Ramesh et al exhibited that the average grain size number is decreases when compared to uncoated Al6061 [20]. The improved grain refinement on coated alloy and the fabricated composites can be mainly allowed to the variable metal formation during coating which is efficiently a thermal process [11].

Figure 12 shows SEM photograph of Al6061 and Ni–Cu–Zn coated aluminium composite. It indicates that there is a good wettability between copper and aluminium interface except some voids and also carbon fibres and copper interface [20]. It can be observed from SEM image that there is no damage for carbon fibres due to which there no much degradation of carbon fibre properties.
Damping measurement

The damping measurement of Al6061 and Ni–Cu–Zn coated Al6061 composite is discussed in this section. The peak values of amplitude of oscillation are noted as shown in the table 4. The obtained values are applied to the standard formula to get the damping of the specimen. It is observed from the damping displacement graphs as presents the decay curve of aluminium composite specimen is decay faster than the uncoated Al 6061 specimen [28]. From calculation damping ratio of aluminium composite is increased by 25% for the applied load compared to Al 6061 specimen. This presents that the damping property of composites increases when reinforcement gets stiffer with matrix material [27].

The calculated damping ratio values for the prepared specimens with the variations in frequency modes are shown in figure 13. It is exciting to observe that the specimen exhibits different damping ratios at the various
frequency modes. Conversely the coated specimen has better damping ratio in the range of 9 KHz to 15 KHz. But in this case, the average damping ratio for frequency mode is 0.07363, mode 2 is 0.06152, and mode 3 is 0.05343, but mode 4 has significant variation among damping ratios i.e., 0.04127. From these results concluded that the strength level is increased due to reducing oxidation by coating thin layers aluminium composite. Chandra et al reported that due to the addition of nickel zinc coating increasing in damping ratio proves this as structural damping material. As the percentage maximizes the stiffer level of composite at higher frequencies which proves Ni–Cu–Zn addition to be suitable reinforcement element satisfying the requirement of a carbon fibre reinforced composite [25]. The dynamic behaviour of Al$_2$O$_3$ reinforces aluminium composites concluded that, the dynamic properties of nano composites were improved by increasing the volume fractions of nano particulates and decreasing the nano particulates size. The results also indicated that, the damping factor, and the related parameters was strongly affected by increasing both volume fraction and the particulates [27]. The damping capacity of SiC and graphite reinforced composites has a greater difference in coefficient of thermal expansion between the Al alloy and the SiC particles. It was found that the higher damping capacity exhibited by this composite in comparison to steel reinforced composite compositions produced [29]. The decrease in storage modulus with increase in temperature is attributed to the decrease in the dynamic stiffness of the composites with temperature. The damping capacity of the composites, as with loss modulus, generally increased with increase in the test temperature effect of the test frequencies 5 Hz and 10 Hz on the damping properties was on the average marginal, while significant variation in damping properties with test temperature were observed in the study [30].

**Conclusions**

In this study, the growth of Ni–Cu–Zn electrolyte coated carbon fibre reinforced aluminium composite is studied and the mechanical and microstructure properties of coated composite are evaluated. The important conclusions are as follows:

1. The Ni–Cu–Zn coated composite showed an improved tensile strength and deformation ability. This is achieved by proper reinforcement of carbon fibres and quantity of fibres. The Cu–Zn coating prevents the formation of aluminium carbide by formation of stable aluminium copper intermetallic compound during casting which avoids contact between molten metal and carbon fibre.
(2) The hardness of coated composite slightly increases due to tough grain refinement of carbon fibres formation. The elongation of cast Al6061 alloy and Ni–Cu–Zn coated Al6061 composites are 6.9% and 7.4% respectively. Increase in the reinforcement of carbon fibre in Al6061 there is a decrease in the density and increase in porosity.

(3) The microstructure study presents Ni–Cu–Zn coated composite with emphasis on minimizing the reduction in carbon fibre strength due to oxidation in presence of the hot aluminium matrix. This decreases the structural defects of coated Al6061 composite.

(4) Damping property of the material is high. Damping property increases when reinforcement gets stiffer with matrix. The damping ratio of Ni–Cu–Zn coated Al6061 composite is increased by 25% for the applied load compared to uncoated Al6061. This will provide good heavy duty damping applications.

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