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Ni-P가 코팅된 유리섬유와 Al_2O_3 나노선으로 강화된 비닐에스터 복합재료의 기계적-열적 특성

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Ni-P Coated Glass Fiber/Al_2O_3 Nanowire Reinforced Vinyl Ester Composite: Investigation on the Mechanical and Thermal Properties

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Abstract: This study mainly focuses on the evaluation of mechanical and thermal properties of vinyl ester composite incorporated with E-glass fiber and aluminium oxide nanowires. These glass fibers are coated with Nickel Phosphorus (Ni-P) via electroless plating method after many trials. The Ni-P coated glass fiber (GF) increases the mechanical properties of the composite when used as the reinforcement. The maximum level of 53% increase in the tensile strength occurs at 0.75 wt% concentration of aluminium oxide nanowires and 40% of Ni-P coated GF as reinforcement. The similar outcome occurs for the other mechanical and wear properties. The thermal analysis reveals that the thermal stability of the composite increases with the increase in the concentration of Al_2O_3 nanowires as fillers and Ni-P/GF as reinforcement. Scanning electron microscope and optical microscope images are used to analyze the surface morphology of Ni-P coated GF, fractured and worn out surfaces of the samples.

Keywords: hybrid composite, Al_2O_3 nanowire, electroless Ni-P, mechanical, wear, thermal property.

Introduction

Polymer composite materials are basically combination of matrix and fiber as reinforcement. Vinyl-ester resin is one of the most commonly used thermoset resins in polymeric composites. Vinyl-ester resin, as a structural polymer, is chosen as a matrix in this study. In fact the cured resins are thermosetting with a network structure has outstanding thermal and mechanical properties. Moreover, it has the ability to withstand high resistance towards moisture and chemicals.[1-3] Glass fiber reinforced polymer (GFRP) composite has extensively been used as a replacement of metals in many applications because of its notable properties such as greater strength to weight ratio, good corrosion resistance, ability to pursuit the production of complex shapes and many more notable properties. GFRP composite has extensively been used in automobile, aerospace, marine industrial, structural and sports applications.[4-7]

Y. Zhang *et al*.[8] have coated the graphite nanosheets by electroless Ni-P and it has been used as the reinforcement. The Ni-P coating over the glass fiber (GF) tends to increase the magnetic property and mechanical property of the prepared composite. Y. Xiang Lu *et al*.[9] had studied the property of the copper coated bamboo fiber reinforced PMC and found that the copper coating increases the electrical conductivity and mechanical property (strength) to an extent.

The incorporation of nanoparticles in the GFRP has gained more importance due to tailor-made properties of the final composite such as increase in mechanical, thermal, electric, electronic and optical properties.[10-12] Last few decades, different spherical shape nanoparticles were extensively used as fillers in the hybrid composite and their influences were studied. These nanoparticles have attained a good attention to the

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researchers due to their very good strength to weight ratio. The addition of nano particles causes agglomeration of particles poses lack of uniform dispersion which can be completely overcome by ultrasonic method.[13-16] Recently, one-dimensional nanoparticles have acclaimed an important role in polymer composite manufacturing. Significantly, the single-wall and multi-wall carbon nanotube added polymer matrix composite are extensively developed and their impact in the mechanical, wear, thermal properties were examined and studied in detail. The properties were found to be improved significantly after the incorporation of these nanomaterials in the composite. Even very small addition of these nanoparticles has a great effect on the mechanical and other properties of the prepared composites.[17-20]

Hence in this study, the electroless Ni-P coated GF and aluminium oxide nanowires are used as the reinforcement and filler in polymer composite. Hand lay-up technique assisted with compression moulding is used to prepare the fiber reinforced composite with the addition of aluminium oxide nanowires at different loading. The mechanical, wear and thermal properties of the hybrid composites are analyzed and also elaborately discussed.

Experimental

Materials and Methods. E-Glass fiber bought from Saint-Gobain glass pvt. limited, India is used as reinforcement. An epoxy-based vinyl ester resin (Derakane 510A-40 bisphenol) with 1.23 g/mL and 38% of density and styrene content is used as the matrix material, methyl ethyl ketone peroxide as catalyst, cobalt octovate as accelerator and dimethylasiline as promoter (procured from Sigma Aldrich, United States). The Al_2O_3 nanowires of diameter 2-6 nm and length 200-400 nm were procured from Sigma Aldrich, United States is used as filler material.

Ni-P is coated over the GF by means of electroless coating process. After many trial and errors, the best bath composition is arrived for proper electroless plating process. The substrate should be thoroughly cleaned and pre-treated ahead of electroless coating process. Ethanol is used for cleaning the substrate followed by etching it in sodium hydroxide solution for about an hour. The GF to be coated is a non-conducting material, hence it is essential to activate the GF surface by means of palladium chloride (from Alfa Aesar, US) solution. Between each pace, the GF substrate should be rinsed with distilled water. The electroless plating bath consists of a source for Nickel (Nickel Sulfate), reducing agent (Sodium Hypophosphite), stabilizer (Tri-sodium citrate) and regulator (Ammonium Chloride) at predetermined quantity for proper coating process. The pH value of the bath is maintained constant by adding ammonia solution at regular intervals. Hot plate with PID controller is used to heat the electrolyte bath. The schematic representation of the hot plate is shown in Figure 1. When the operating temperature of 75±2 ºC is reached, the GF is placed in the bath for coating. The pH value of the electrolyte solution should be maintained between (7-8) pH for proper coating process.[21] In Table 1, the composition of bath chemicals used in electroless solution is clearly given. The formation of the Ni-P particles by electroless coating process is explained below by the following eq.:

$$H_2PO_2^- + H_2O \rightarrow H_2PO_3 + 2H^+ + 2e^-,\ E^0 = 0.50\ V \quad (1)$$

$$Ni^{2+} + 2e \rightarrow Ni^0,\ E0 = -0.25\ V \quad (2)$$

$$2H^+ + 2e \rightarrow H_2,\ E^0 = 0.000\ V \quad (3)$$

$$H_2PO_2^- + 2H^+ + e \rightarrow P + 2H_2O,\ E^0 = 0.50\ V \quad (4)$$

The electroless nickel-phosphorus plating process consists of oxidation and reduction reaction. From eq. (1) it is clear that the hypophosphate oxidizes to orthophosphate and liberate number of free electrons. These free liberated electrons reduce the nickel ions from the electrolyte solution to nickel particles in eq. (2) and liberate the hydrogen in the form of bubbles from the electrolyte solution as per eq. (3). The excess amount of free liberated electrons reduces the sodium hypophosphate to phosphorus particles and water in eq. (4). The reduced nickel and phosphorus particles from the electroless solution deposits over the palladium chloride activated GF substrate.

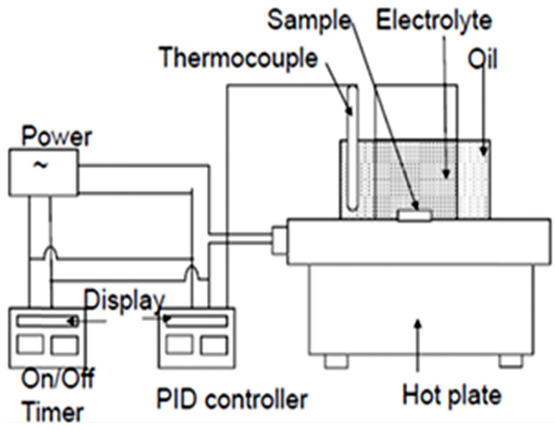

Figure 1. Electroless coating process setup.

Table 1. Electrolyte Bath Composition

EN bath compositions	Quantity (g/L)
Nickel sulfate[a] ($NiSO_4$)	40
Sodium hypophosphite[b] ($NaPO_2H_2$)	20
Sodium tricritrate[c] ($Na_3C_6H_5O_7$)	50
Ammonium chloride[d] (NH_4Cl)	60
Temperature	75 °C (±2 °C)
pH	7-8

[a]Nickel source. [b]Reducing agent. [c]Stabilizer. [d]Complexing agent.

Proper activation plays a major role in the plating process.

The composites of dimension 200 mm×100 mm are prepared by hand-layup technique followed by compression moulding and allowed to cure for 5 h at room temperature. Initially, the resin is mixed with the functionalized nanowires using mechanical stirring followed by ultrasonic stirring to ensure uniform distribution. The Ni-P coated GF and Al_2O_3 nanowires are used as reinforcement and fillers respectively in the hybrid composite. In this work, ten different materials are fabricated and their properties are evaluated and studied. The sample identity with their various compositions is tabulated in Table 2.

Testing Procedure. Mechanical Properties: The tensile test is carried out by Instron universal testing machine (Model 4301) which available at CIPET, Chennai, India. The samples are prepared as per ASTM D638 with a crosshead speed of 2 mm/min. The universal testing machine of model Shimadzu AG-IS 50KN is used for performing the three point bend test. The samples for flexural strength are prepared as per ASTM D790 with a crosshead speed of 3 mm/min. The impact strength is examined by digitalized Izod Pendulum impact tester. The samples for the impact test are prepared as per ASTM D256. The values are arrived through the average of five tested specimens.

Wear Analysis: Pin on disk apparatus is used for analyzing the wear property of the fabricated composite with ASTM G99 standard. The specimen is prepared as a pin of diameter 10 mm and height 25 mm. The tests are performed at fixed conditions such as load, velocity and sliding distance of 20 N, 0.5 m/s and 1000 m. The initial and final weights are noted. The volume loss of the composite is calculated from the weight loss using the formulae given in eq. (5):

$$Volume\ \ loss = \frac{[Mass\ loss\ in\ gm \times 1000]}{Density\ of\ the\ composite}\ \mathrm{mm}^3 \quad (5)$$

From the volume loss the specific wear rate can be determined using the formulae given in eq. (6):

$$Specific\ \ wear\ \ rate = \frac{Volume\ loss}{Load \times Sliding\ distance}\ \mathrm{mm}^3/\mathrm{Nm} \quad (6)$$

Thermogravimetric Analysis: The thermal degradation behavior of the prepared composite is performed by using TGA-DTA/DSC equipment make of TA instruments model Q600 SDT. For thermal analysis, powdered sample of 10 mg is taken in a platinum pan and heated at 5 °C/min till it reaches 600 °C. The test was carried out in the nitrogen atmosphere.

Microstructure Analysis: SEM and FE-SEM micrographs were used to study the surface morphology of the Ni-P coated GF and the fractured surface of the composite specimen. The instrument used for taking micrographs is Hitachi S3400N model and field emission scanning electron microscope of

Table 2. Sample Identity with its Composite Composition

Sl. No.	Sample identity	Material composition			
		Vinyl ester resin (wt%)	Glass fiber (wt%)	Ni-P Coating (wt%)	Nanoadditives (wt%)
1	10BGF	90	10	0	0
2	11 / 0	89	10	1.0	0
3	11 / 0.25	88.75	10	1.0	0.25
4	11 / 0.50	88.50	10	1.0	0.50
5	11 / 0.75	88.25	10	1.0	0.75
6	11 / 1.0	88	10	1.0	1.0
7	22 / 0.75	77.25	20	2.0	0.75
8	33 / 0.75	66.25	30	3.0	0.75
9	44 / 0.75	55.25	40	4.0	0.75
10	55 / 0.75	44.25	50	5.0	0.75

model JSM 6701F. The samples are coated with gold before subjected to SEM analysis. EDAX analysis is performed in SEM with oxford EDAX analyzer. Optical microscope with image analyzer is used to study the worn out areas of the wear surfaced sample.

Results and Discussion

Characterization Study. The SEM micrographs are used to study about the surface morphology of the GF. The SEM micrographs with different magnifications of both bare GF and nickel coated GF are shown in Figure 2. The Figure 2(a) and (b) shows the micrograph of un-coated GF having a smooth surface with some impurities present over it. These impurities can be removed by the proper pre-treatment process. The Figure 2(c), (d), (e) and (f) shows the SEM and FE-SEM micrograph of the GF coated with electroless Ni-P at lower and higher magnifications. The presence of spherical shaped particles indicates the Ni-P coating over the substrate.

EDAX or quantitative analysis is carried out to study about the presence of element present in the sample. It gives us a clear idea of the quantitative concentration of the elements present over the substrate. The Figure 3(a) and 3(b) reveals that silica and nickel are the major trace element in the bare and Ni-P coated GF.

Tensile Strength. Figure 4 shows the tensile strength of various hybrid polymer composites. The plotted results are the average value of 5 tested specimens for each composition. From the Figure 4, it can be inferred that the tensile strength and modulus of the composite increases from 41.92 to 45.30 MPa and 1174.97 to 1494.22 MPa (i.e. 8% and 27%)

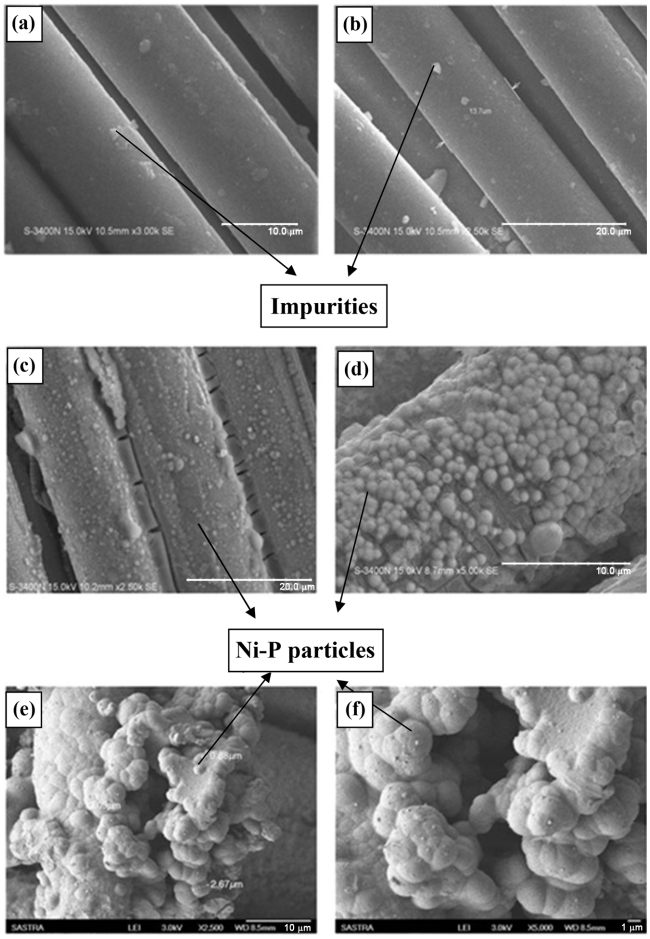

Figure 2. SEM micrograph (a) & (b) bare glass fiber; (c) & (d) Ni-P coated glass fiber at lower and higher magnification; (e) & (f) FE-SEM micrograph for Ni-P coated glass fiber at lower and higher magnification.

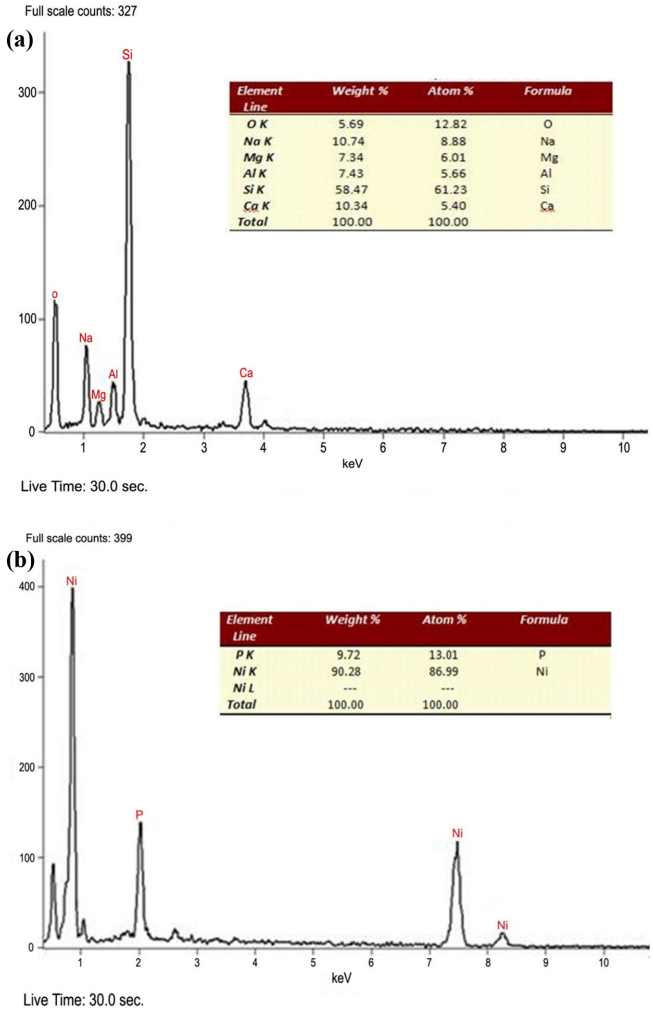

Figure 3. (a) EDAX graph for the uncoated glass fiber; (b) EDAX graph for the nickel coated glass fiber.

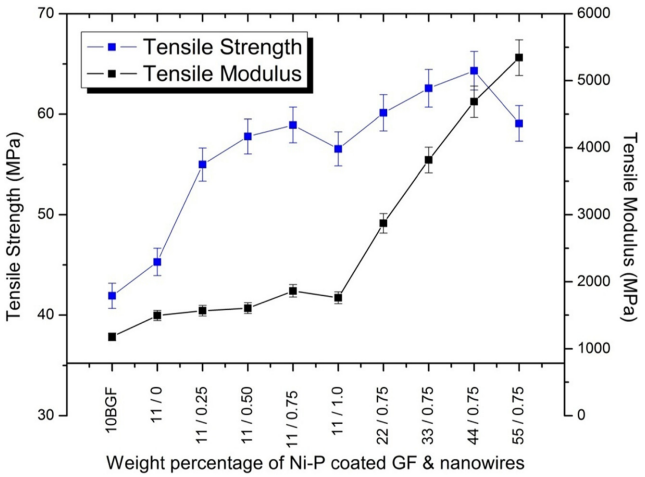

Figure 4. Tensile strength variation of the hybrid composites.

respectively when 11% Ni-P coated GF is used as the reinforcement. The surface of the glass fiber is modified with Ni-P particles.[22] This increase in the strength is due to the presence of Ni-P particles over the GF. The Ni-P particles present over the GF has a good adhesion with it and also act as the load carrying member in the composite during the tensile loading which helps to sustain more load than bare GF reinforced composite. Now keeping 11% Ni-P coated GF as constant, the aluminium oxide nanowires are added at different concentration. The maximum tensile strength and tensile modulus of about 58.93 and 1862.59 MPa are obtained when 11% Ni-P coated GF + 0.75% Al_2O_3 is used as reinforcement. This is due to the fact that the nanowires which are evenly distributed in the matrix and adhere to it firmly and once the crack is initiated on

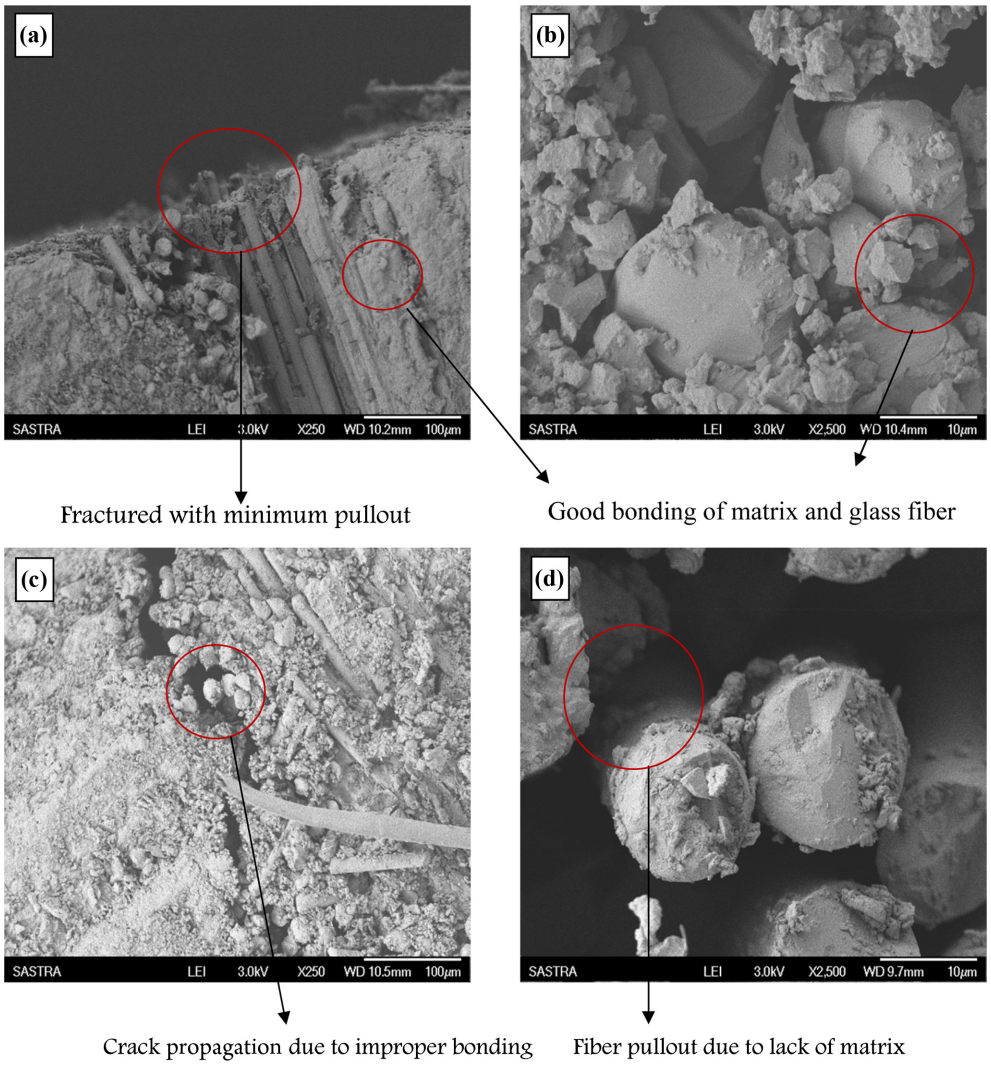

Figure 5. (a) & (b) FE-SEM Micrograph of fractured surface of 44%Ni-P/GF 0.75 Al_2O_3 reinforced composite (c) & (d) micrograph of fractured surface of 55% Ni-P/GF 0.75 Al_2O_3 composite.

the surface of the composite due to uniaxial load these nanowires arrest the propagation of the cracks and withstand the load to an extent without failure. But with further increase in the concentration (1% weight) of the nanowires, the strength and modulus drop to an extent due to two main causes: (a) The increase additions of fillers lead to non-uniform dispersion and agglomeration of the nanowires in the composite. (b) The other reason is that by adding more quantity of filler to the matrix, the viscosity increases and therefore the possibility of air traps and the introduction of microvoids are enhanced. Hence the microvoids and agglomeration in the composite increases the stress concentration.[19]

This agglomeration of nanowire affects the amorphous nature of the polymer composite which leads to sudden rupture at lower loads. This is the outcome anticipated and is in accordance with the literature data.[20] Hence 0.75 weight percentage of Al_2O_3 nanowire is fixed as the optimum level. The amount of reinforcement (GF) used is increased from 11 percentage to a higher concentration. When the amount of Ni-P coated GF is increased then simultaneously there is an increase in the tensile strength and tensile modulus to a greater extent. The maximum tensile strength and tensile modulus of 64.32 and 4685.71 MPa are observed from the material composition of 55.25% matrix, 44% Ni-P/GF reinforcement and 0.75% Al_2O_3 fillers reinforced composite. This high strength occurs because the laminate arrangement of the Ni-P/GF and Al_2O_3 nanowire resist the initiation of crack to pass through it and further carries more loads during uniaxial tensile loading.

The nanowires are cross-linked with the GF and hold it firmly during loading condition to transfer more loads and avoid slipping or pullouts of GF which can be clearly seen in Figure 5(a) & (b) and hence maximum strength and modulus are obtained. Further, when the concentration of the Ni-P/GF to 55%, the adhesion between GF and the matrix reduces and pull out of fiber occurs easily which is clearly seen in Figure 5(c) and (d) which leads to the drop down in the strength of the composite.

Flexural Strength. The flexural strength of the fabricated vinyl ester composite was determined using three point bend test. For each specimen, the tests were conducted and the results were plotted as an average for 5 tested specimen. Figure 6 shows the plot of flexural strength of hybrid polymer composites with a mixed trend compared to tensile strength. The flexural strength of composite with bare GF is about 77.20 MPa and increases when Ni-P coated GF is used as the reinforcement to 82.25 MPa. But with the addition of small

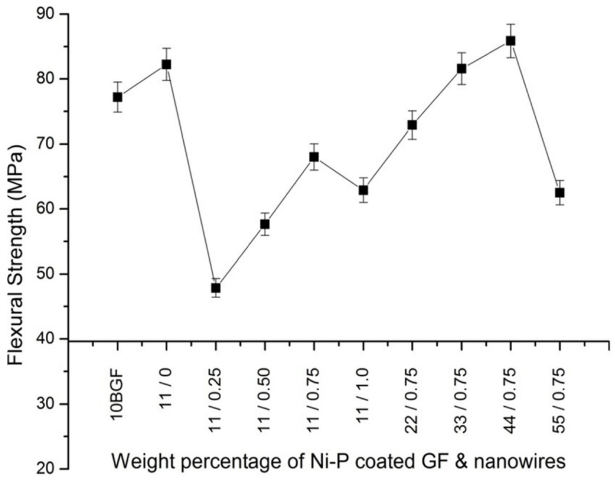

Figure 6. Flexural strength variation of the hybrid composites.

amount (0.25 wt%) of Al_2O_3 nanowires to the composite, the flexural strength drops suddenly to 47.85 MPa, with the addition of increased amount of these nanowires the flexural strength increases to an extent. This is due to the phenomenon called crazing. Crazing is a microscopically localized phenomenon which involves a large degree of localized plastic deformation. The craze is different form of crack that it cannot be felt at the surface. The craze in the materials will continue to support the load with the microvoids and nano level polymer fibrils in it inside the surface. Because at this low-level concentration the nanowires in the PMC acts as nanoscale stress inducers and initiates stress during flexural loading. But at higher concentration of these nanowires, they are likely to form a continuous chain like or closely packed network structure which arrests the enlargement of cavitations and prevents the composite to break at lower flexural loads. The maximum flexural strength of about 68 MPa is observed when Al_2O_3 nano wire of 0.75% is added to the matrix as fillers after which again there in a drop in the flexural strength at 1% inclusion of the nanowires which may be due to the agglomeration of the nanoparticles as said earlier. Similar kind of result was also perceived by Daniel *et al*..[17] Similar to the tensile behavior, 0.75 wt% of nanowire is kept as constant and the amount reinforcing material (Ni-P/GF) is increased from 11% to 55%. As the amount of reinforcing material increases, the flexural strength also increased and a maximum of 85.86 MPa is observed for 0.75% Al_2O_3/40%Ni-P GF reinforced composite. This increase in the flexural strength is because of the reinforcing material. When the reinforcing materials are placed over one another in the form of laminates it withstands a max-

imum flexural load given to the specimen without craze or crack initiation and even if the crack is initiated it avoids the propagation of the crack through its surface and ensures a maximum strength. But due to the improper adhesion at 50 wt% concentration of the Ni-P/GF, the strength decrease.

Impact Strength. The influence of Ni-P/GF and Al_2O_3 nanowire on the impact strength is shown in Figure 7. The plotted values of the impact strength are the average of 5 tested specimens for each sample. The un-notched specimen is held as a vertical cantilevered beam and is broken by a sudden high load given by pendulum.

The impact strength of the uncoated GF reinforced polymer matrix is noted as 25.63 kJ/m^2 and it tends to increase up to 3% when Ni-P coated GF is used as the reinforcement. But with the addition of the Al_2O_3 nanowires in the matrix at different concentration (0.25, 0.50, 0.75, 1.0 wt%), it has a reverse effect on the impact strength of the composite. This phenomenon is inevitable because the composite gets hardened and in brittle nature with the addition of nanoparticles. Due to this outcome, the impact strength decreases gradually with increase in the filler concentration. When the load is applied at high strain rate to the specimen, these nanowires present in the composite acts as the stress raisers and make the final composite to fracture suddenly without absorbing more energy.[17] But there is a sudden increase in the impact strength when the percentage of reinforcing fiber increases. The maximum strength of 71.79 KJ/m^2 is observed for 44% Ni-P/GF 0.75% Al_2O_3 reinforced polymer composite. This is because the increases in the concentration of Ni-P/GF layer have the ability to absorbs more energy during the sudden load and also increase the impact strength of the composite. The bonding of Ni-P/GF, Al_2O_3 nanowire and the matrix should ensure good adhesion with them. The impact fractured composite reinforced with more wt% Ni-P/GF shows a fiber pull-out mechanism and total rupture of the fiber and matrix after the impact test.

Wear Analysis. Figure 8 clearly indicates the specific wear rate of the different composites. The addition of nanowires and Ni/P/GF has a positive influence on the wear resistance of the composite. The 10% bare GF reinforced polymer composite has a maximum wear rate of 0.897×10^{-5} mm^3/Nm and it is clear from the Figure 9(a) and 9(b) the wear tracks which is parallel to the sliding direction have deep grooves or scratches on the worn surfaces which suggest that the specimen had undergone severe adhesive and abrasive wear. The specific wear rate decreases with increase in the concentration of Al_2O_3 nanowire. This is because the nanowires dispersed evenly in the matrix adhere with it firmly and do not allow the particles on the surface to wear out easily. At 0.75 wt% concentration of Al_2O_3 nanowire, the wear rate decreases to 0.637×10^{-5} mm^3/Nm whereas at 1.0 wt% concentration of Al_2O_3 nanowire there is step-up in the wear rate which is due to the agglomeration of the nanowires. Figure 9(c) and 9(d) clearly shows the wear track, on which the worn out surfaces have more cavities due to the removal of the cluster of nanowires formed by agglomeration nature which leads to increase in specific wear rate. These nanowires which are agglomerated in the composite pulls out easily by action due to weak adhesion with the matrix and cavities are formed along with the deep groves during sliding

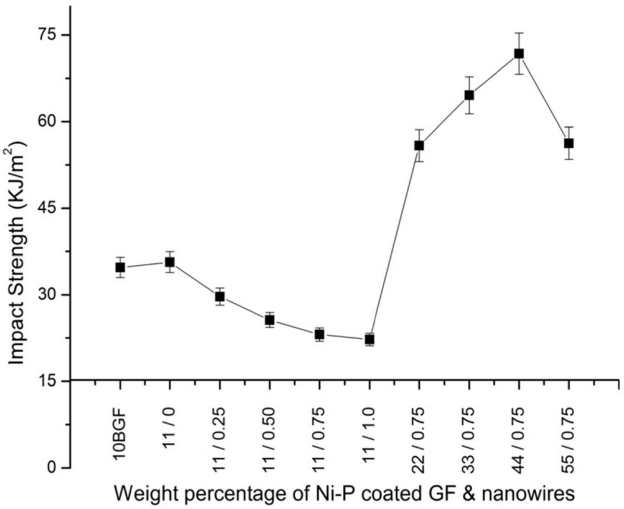

Figure 7. Impact strength variation of the hybrid composites.

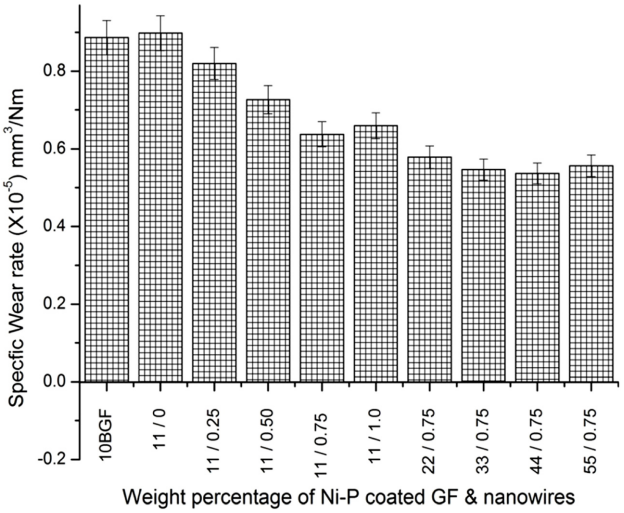

Figure 8. Specific wear rate variation of the hybrid composites.

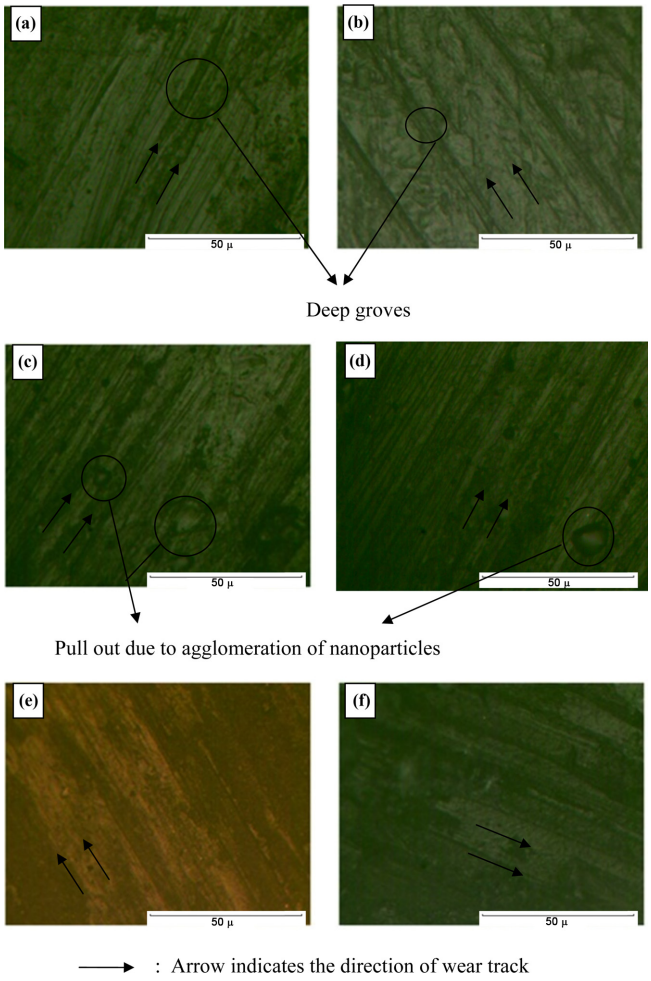

Figure 9. Optical microscope images of the worn surfaces (a) & (b) 10 wt% GF reinforced composite; (c) & (d) 11 wt% Ni-P/GF/ 1.0 wt% Al_2O_3 nanowire reinforced composite; (e) & (f) 44 wt% Ni-P/GF/0.75 wt% Al_2O_3 nanowire reinforced composite.

wear. Figure 9(e) and 9(f) shows the worn out surface of the composite with 0.75 wt% Al_2O_3 nanowires and 44% Ni-P/GF reinforcement. The worn surface parallel to the wear track has with very minimum scratches over it and those scratches are not in-depth as discussed earlier in this section. This is due to the fact that in this composite the wear detritus formed makes a thin layer on the mating surface during sliding which almost acts as the solid lubricant and minimum specific wear rate of 0.536×10^{-5} mm^3/Nm is observed for the composite sample.

Thermogravimetric Analysis. The thermal stability of the hybrid polymer composite is analyzed in thermogravimetric analyzer (TGA). The test is conducted with all the samples and the results were analyzed. Figure 10 shows the thermograms of the bare GF and other composite reinforced with Ni-P/GF and nano Al_2O_3 wire.

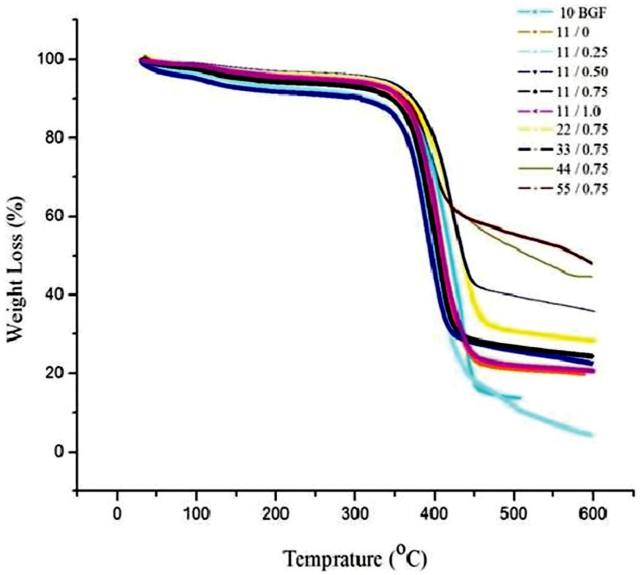

Figure 10. Thermogravimetric analysis (TGA) curves for the different hybrid composites.

From the result, it is clear that the thermal stability of the composite increases when coated GF is used as the reinforcement and further increases with the increase in the concentration of Al_2O_3 nanowires and Ni-P/GF in the matrix. A trend similar to this was reported by Joshi *et al*..[23] From the thermogram, it is noted that there are three stages of decomposition occurs during TGA. In the first stage, the loss is minimal due to the evaporation of moisture content present in the sample. In the second stage of decomposition, the bare GF reinforced composite starts to decompose rapidly from 325 °C and ends with 2% residue content. The start temperature of the second stage of decomposition significantly increases with the addition of Al_2O_3 nanowires and Ni-P/GF which indicates the increase in thermal stability of the composite which is clearly witnessed in Figure 10. The temperature at which the utmost degradation takes place is increased by 50 °C and ends up with an increase in the residue content of about 50 wt%. This is because the nanowires and the Ni-P/GF in the composite which are evenly dispersed results in good interfacial bond strength with the matrix. This arrest the movement of polymer chains at increased temperature[10] and thus enhance the thermal stability of the hybrid composite.

Conclusions

In this work, the optimum bath composition for electroless nickel-phosphorus coating over the GF substrate was deter-

mined. The SEM micrograph of the coated GF clearly shows the presence of dense Ni-P particle over the surface of the GF. The mechanical properties and the thermal properties have a positive response when coated GF is used as the reinforcement.

The tensile strength of the hybrid composite increases with the Ni-P coated GF is used as the reinforcement to 8% and further enhances with the increase in concentration of Al_2O_3 nanowires and Ni-P coated GF. The maximum increase in the tensile strength of about 53% is achieved for the composite with 44 wt% of Ni-P/GF and 0.75 wt% concentration of Al_2O_3 nanowire. For the case of flexural property, initially, the flexural strength decreases with the addition of small amount of nanoparticles and then increases gradually with the increase in the filler material and the reinforcement.

The specific wear rate also decreases with the addition of filler material and the reinforcement. The thermal stability of the composite increases when Ni-P coated GF is used and further increases with the increase in the concentration of nano additives and Ni-P GF as reinforcement.

Hence, the nickel-phosphorus coating over the GF surface using electroless method with the addition of Al_2O_3 nano wire as fillers improves the mechanical, thermal and specific wear rate of the hybrid composite which makes them as a suitable for medium temperature structural applications.

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