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INVESTIGATION OF THE EFFECT OF INCONEL PARTICULATE FILLER ON THE MECHANICAL AND FIRE-RETARDANT CHARACTERISTICS OF GFRP COMPOSITES

RAZISKAVA VPLIVA DODATKA POLNILA NA OSNOVI DELCEV IZ INCONELA NA MEHANSKE IN OGNJEVARNE LASTNOSTI GFRP KOMPOZITOV

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This paper reports the effect of incorporating Inconel particles as filler for enhancing the delamination and mechanical strength of glass-fiber-reinforced epoxy composites. It is intended for application in renewable energy. Glass fiber plays a vital role in renewable-energy industries for its non-corrosiveness and lower maintenance cost. The mechanical properties of glass fibers make the renewable industry for effective utilization in the manufacturing of turbine blades in onshore and offshore environments, as well as the addition of fillers enhances the mechanical properties of the material. This study comprises validating three different compositions of Inconel blended with epoxy resin for the preparation of laminates using a vacuum-infusion process. The tensile and flexural properties of the composites were experimentally examined and validated for the enhanced use of structural applications in the field of renewable energy. In addition to the mechanical characterization, a finite-element validation was used to determine the delamination effect. The Ansys Composite PrePost (ACP) module was used to validate the Inconel-blended composite materials. The flammability characteristics were determined as per the UL-94 standard for both vertical and horizontal flame tests. The water-absorption characteristics were also estimated for the three different proportions of the Inconel-filled laminates. The study reveals that the incorporation of Inconel powder enhanced the mechanical properties, contact angle and the fire-retardant characteristics of the glass-fiber epoxy-blended laminates.

Keywords: delamination, Inconel, flammability, mechanical properties

V članku je opisan vpliv dodatka delcev Ni superzlitine Inconel k, s steklenimi vlakni, ojačanemu epoksidnemu kompozitu (GFRP; angl.: Glass Fibers Reinforced Particle epoxy composite). Namen dodatka je bil izboljšanje odpornosti kompozita proti cepljenju in povečanje njegove mehanske trdnosti. Steklena vlakna imajo močno vlogo v industriji naprav za obnovljivo energijo (npr. lopatice v vetrnih elektrarnah) zaradi dobrih antikorozijskih lastnosti in majhnih stroškov vzdrževanja. Mehanske lastnosti pletiv iz steklenih vlaken v komponentah za obnovljivo energijo omogočajo izboljšanje učinkovitosti turbinskih lopatic v vseh okoljih (morje, kopno), ki pa se z dodatnimi polnili kompozitnega materiala še izboljšajo. V študiji so ocenjevali lastnosti laminatov izdelanih z vakuumskim nalivalnim postopkom. Pri tem so uporabili tri različne sestave oziroma mešanice epoksi smole z delci Inconela nalite na pletivo iz steklenih vlaken. Eksperimentalno so določili natezno in upogibno trdnost kompozitov in jih ocenili za izbrane konstrukcijske oziroma funkcionalne elemente, uporabne na področju obnovljive energije. Poleg mehanskih lastnosti so ocenili še delaminacijo s pomočjo metode končnih elementov. Uporabili so programsko orodje Ansys z modulom Composite PrePost (ACP). Vžigalne lastnosti izdelanih laminatov so določili po standardu UL-94 za obe smeri, z vertikalnim in horizontalnim preizkusom. Ocenili so tudi absorpcijo vode (kontaktni kot) v laminatih s tremi različnimi vsebnostmi polnila oziroma delcev Inconela. Ugotovili so, da dodatek delcev prahu iz Inconela izboljša mehanske lastnosti, poveča kot omakanja in odpornost proti vžigu izdelanih kompozitov iz epoksi laminatov ojačanih s steklenimi vlakni.

Ključne besede: delaminacija, Inconel, vnetljivost, mehanske lastnosti

1 INTRODUCTION

In recent years the delamination of composite materials has become a serious issue in many industrial sectors. Composite structures have been designed for a specific requirement and it has been made suitable for use in different environmental conditions. The delamination effect in composites has been focused as the major cause of the damage to structures. The delamination occurs when the inter-laminar stress of the plies exceeds the overall yield stress of the materials interfaced. Delbariani et al. investigated the inter-laminar fracture toughness of laminates based on the critical energy. If the rate of the energy released on the laminates exceeds the critical energy, then the chances of the delamination were found to be a maximum.[1] Dong estimated the effect of process-induced small voids in fiber-reinforced composites also assumed to have an increased axial shear modulus and transverse modulus compared to the large voids. The influence of the voids in the fiber-reinforced composites influences the physical properties of the material.[2] Swolfs et al. validated the flexural behavior of the material, generally the flexural tests were performed using a three-point bending apparatus under bending condition, the upper layers will tend to be in compression mode and the bottom layers in the tension mode, which causes an inter-laminar shear

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and potentially initiates the delamination across the thickness for determining the delamination strength of the materials.[3] Fotouhi et al. determined that the majority of the damage in the composites is due to the fiber matrix de-bonding. He also estimated that mode-1 conditions are found in the unidirectional specimens, compared to the woven fabric specimens. The SEM examinations also confirmed the above results. Further he used acoustic emission to determine the delamination in glass and epoxy-composite materials.[4]

Peng et al. discussed the improvement of material performance by reducing the delamination effect. The geometrical featuring of the composite materials with long fibers provides better longitudinal strength and stiffness, whereas the polymer matrix is responsible for the toughness and delamination strength of the material.[5] Zou et al. focused on the composite planar behavior and the properties, the thickness of the laminate exhibited considerably lower material characteristics. The major failure mode is due to the parting of an initially bonded single ply, causing the delamination. The machining of the composites also causes delamination to occur, and the damage extension criterion was used to determine the delamination. The damage-extension criterion highlights the threshold level of the strain rate, thus making it possible to determine the delamination by altering the cohesive elements attached to the geometrical structure.[6] Hiroakiet et al. used carbon nano-fiber reinforcement in the interlayer of the composite material for preparing mode-I specimens with three different amounts of nano-carbon at the interlayer from 10 g to 20 g the fracture toughness has been improved, but when the amount of non-carbon increased to 30 g, it decreased the fracture toughness of the material. This information provides a valid procedure that a limited addition of reinforcements only provides a structural improvement.[7] Zhao et al. experimented on PTFE (poly-tetra-fluoro-ethylene) with copper, lead and nickel, with 30 % filler content. The addition of the filler mainly focused on the wear characteristics of the material, with PTFE as the base. The addition of the filler content increased the load-bearing capacity of the materials.[8] Zujin et al. explained that the delamination in wind turbines was found mostly in the outer skin, which resulted in catastrophic failure. The different failures of the delamination happened in the internal core materials, which were found to be in the staggered shape. Due to the deformation, the direction reverses in the condition that the shell of a blade causing it to deform in the reciprocating direction. The trailing edge was considered to be one of the instability zone in the wind turbines due to the large amount of deformation.[9] Y. J. Liu et al. formulated a FE model for determining the impact influenced delamination for fiber-reinforced plastic materials. The paper mainly valued the stacking sequence of the laminates; the laminates with increased adjacent layers showed reduced delamination area. The impact strength of the material can be increased with the proper orientation of the laminate angle with an increased layer angle.[10] Wilk demonstrated the testing methodology for determining the inter-laminar delamination in laminates. The strain energy release rate (SERR) has been defined as the energy required to extend the crack to an infinitesimal length. The mathematical equation representing the SERR δ_R is given by Equation (1).

$$d_R = \frac{P^2 \mathrm{d}C}{2b \cdot \mathrm{d}l} \tag{1}$$

where P is the applied force, b is the specimen width, l is the crack length, and C is the specimen compliance. The three modes of crack propagation are Mode I (opening), Mode II (sliding shear) and Mode III (tearing shear).

The cyclic loading conditions were usually described by the power law relationship between the rate of delamination growth with fatigue cycles to the maximum applied SERR, named Paris' law, and is given by Equation (2).

$$\frac{\mathrm{d}a}{\mathrm{d}N} = \alpha \cdot G_{\max}^{\beta} \tag{2}$$

where $\mathrm{d}a/\mathrm{d}N$ is the delamination growth rate, $G_{\max}$ is the maximum SERR in cycle, and α and β are the experimentally obtained parameters.[11] Finite-element analyses (FEA) were applied for the validation of the structural capacity of the laminates with different length scales. As per the FEA, delamination was predominately identified in the two layers of dissimilar fiber orientation, causing the fiber to peel out from the matrix and the interfacial cracks were also generated on the surface due to a reduction in the stiffness of the corresponding interfacing layer. Cortes et al. concluded that Inconel 718 grade material had the slowest crack growth rate due to its high yield stress, which leads to the development of the plastic zone for avoiding the crack growth.[12] Nikhil et al. evaluated that the reduction in metallic fillers in composites diminished the compressive behavior and improved the impact strength. The key factor for the results was due to the aspect ratio of the filler used in the composite material.[13] Pincheria et al. validated that the interfacial shear strength (IFSS) between the matrix and the fiber determines the internal ply behavior of the composite material and the overall inter-laminar delamination strength of the composites mainly depends on the fiber-matrix interface.[14] Yang et al. claimed that the peel strength and the flammability characteristics are much higher for epoxy-based resins compared to other phenolic resins.[15] The failure in the composite structures was mainly due to the delamination effect in the connecting zones, the improper filling of the resin and the resin deficiency in the maximum load concentrated areas initiates the failure and making the entire structure collapse due to the effect of cyclic loads. The overall reviews encapsulate the selection of filler and the proportions for effective utilization of the materials for specific applications. Inconel 718 grade filler is used for the current study with glass fibers and epoxy resin.

2 EXPERIMENTAL PART

The experimental procedure includes the manufacturing of the laminate by a vacuum-infusion process (VIP) and the mechanical properties of the prepared samples were tested and evaluated to determine the effect of adding the filler material.

2.1 Materials

Araldite LY 556 resin and the hardener Aradur HY 951 were used for preparing the matrix. The E-glass fiber mat was used as reinforcement for the preparation of the samples. The materials were purchased from the Go Green India Pvt limited. As per the supplier, the resin exhibits low viscosity and excellent water resistance. The materials used for preparing the samples are listed in **Table 1**.

Table 1: Materials used for preparing the samples

Description	Material	Grade/Composition
Fiber	E – glass fiber	300 GSM
Matrix Resin	Resin – Araldite Hardener – Aradur	AY-556 HY-951
Filler	Inconel	718

2.2 Chemical composition of the Inconel 718

The epoxy and the glass fibers were used as the matrix and the fiber for the preparation of the composite materials. The Inconel 718 filler of three distinct percentages was added, the general chemical composition of the Inconel filler is shown in the **Table 2.** The size of the Inconel particles was 100 μm and it was spherical in shape.

2.3 Material preparation

The samples were prepared by the VIP shown in **Figure 1**. The main scope of the VIP is to absorb the excess resin in the fiber and to avoid the air traps present during the fabrication of the mat, thus making the material more stable with a resistance to crack propagation. The vacuum-suction pressure is the key source for the VIP, the major aspect of the vacuum infusion process is that the pressure used to drive resin into the laminate for preparation of the components. Three different percentages of Inconel powder were segregated into three different weights for mixing it with the epoxy resin. Initially the

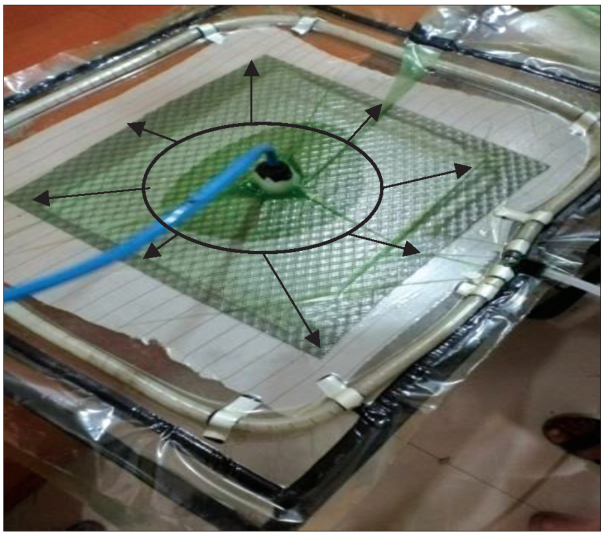

Figure1: Distribution of the epoxy resin under the vacuum-infusion process (VIP)

fiber, resin and the hardener were weighed as per the rule of mixture. The process was conducted as per the procedure. The pump is continuously operated for about 3–4 h, ensuring the air traps are completely relieved from the fiber stacks. The laminate is then allowed to cure for 15–20 h and then it is removed from the glass table and it is kept inside a weave with a temperature of 60 °C to avoid the atmospheric moisture in the specimens.

2.4 Inter-laminar fracture toughness of unidirectional fiber-reinforced polymer composites

The test method describes the Mode-I inter-laminar fracture toughness. The test sample was prepared using unidirectional glass fibers with 0° orientation. The specimen dimension is maintained as 125 mm length, 25 mm width and a thickness of 3 mm. The test samples were conditioned in an oven for 2–3 h to remove the moisture. The tests were conducted at 23 °C.

2.5 Tensile and flexural characteristics

The tensile and the flexural tests were carried out using ASTM D3039 and ASTM D790. The specimen dimensions of (175 × 25 × 2) mm for tensile and the gauge length are maintained as 115 mm and (125 × 12.7 × 3) mm for flexural test. The tensile and the flexural behaviors of the material were estimated for determining the yield stress and the modulus of elasticity of the composite material with the addition of filler. The obtained values were incorporated in the FEA for identifying the delamination characteristics of the material in different loading conditions in the analysis software. The tensile

Table 2: Chemical compositions of the Inconel 718 filler (w/%)

Element	Ni	Cr	Fe	Nb	Mo	Ti	Co	Al	Mg	Si
Chemical composition	50.00–55.00	17.00–21.00	Balance	4.75–5.5	2.80–3.30	0.65–1.15	≤ 1.00	0.20–0.80	≤ 0.35	≤ 0.35

test was carried out for four different groups of the filler. A 100-t servo hydraulic UTM machine was used to execute the testing. The cross-head speed was maintained at 1mm/min to evaluate the tensile strength of the composite material.

2.6 Finite-element validation for determining the delamination effect on the samples

The composite samples were designed using the Ansys design modeler 19.1. The material input data were used for the delamination analysis. The section of the prepared sample was spliced into two equal halves for maintaining a displacement in the solver module for determining the delamination force. The glass fabric was selected in the fabric selection tab and the element sets were generated with the three element sets were generated namely open interface, total interface and total element. The oriented element sets were also prepared for the top, bottom and the open interface. **Figure 2a** shows the sequential ply orientation from the top to the bottom and **Figure 2b** shows the interface delamination zone. The entire setup was validated and the results were iterated with the static structural modular with the fracture and module ply interface delamination. The setup was initiated with the specific constraints for the loading operations for determining the delamination effects in the fiber and the matrix. Gu et al. explained the application of the FEA for determining the delamination in the matrix, particularly at the free edges. The current finite-element validation focus on the interfacial crack in the laminates for determining the delamination characteristics of the Inconel particulate filled material.[16]

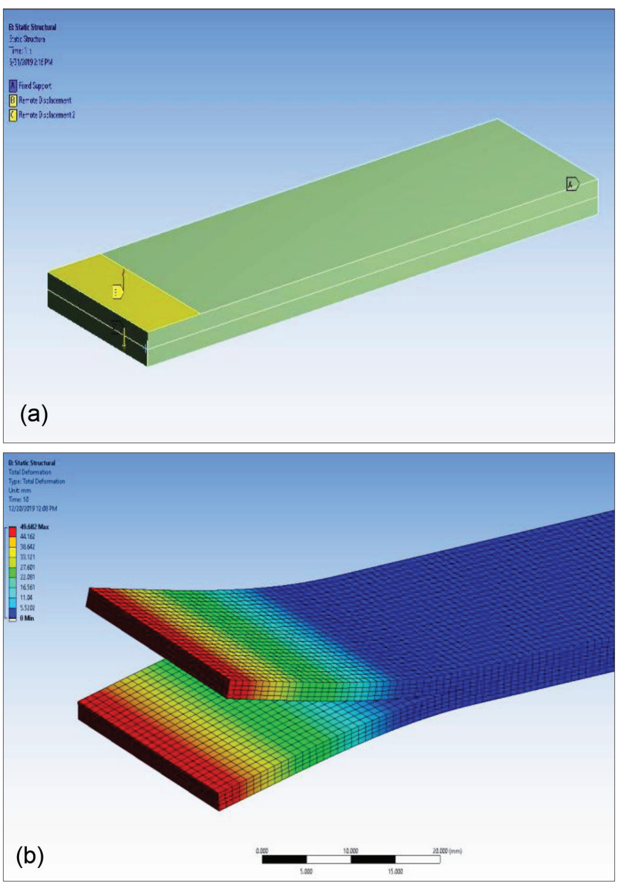

Figure 2: Delamination test using FEA: a) load constraints, b) deformation

Figure 2a shows the load constraints for performing the deformation analysis of the generated composite material from Ansys – ACP modeler. **Figure 2b** denotes the overall deflection with reference to the applied load from the obtained mechanical data.

2.7 Water absorption

The contact-angle measurement was carried out for the water-absorption characteristics of the Inconel reinforced (glass-fiber-reinforced plastic) GFRP composites. The contact-angle analysis provides information about the material, whether hydrophobic or hydrophilic in nature. The measurement was made with the sessile drop method.

2.8 Flammability test

The flammability characteristics of the Inconel-infused GFRP composites were tested using the UL–94 standard for both the horizontal burn test (HBT) and the vertical burn test (VBT). The results were calculated as per the standard for the burn rate and the type of material based on the specification included in the standard.

3 RESULTS AND DISCUSSION

The tensile results of the samples with Inconel 718 of (1, 3 and 5) *w*/%, shows variable results with progressive increments in the stress values.

Table 3: Tensile properties and flexural modulus of the GFRP with (1, 3 and 5) *w*/% of Inconel 718 as filler

Sample	Ultimate load (kN)	Tensile strength (MPa)	Young's modulus (GPa)	Flexural modulus (GPa)
GF + Epoxy	13.334	209	3.892	14.7690
1 % Inconel + GF	25.121	395	4.865	19.1371
3 % Inconel + GF	22.987	362	4.447	12.7112
5 % Inconel + GF	17.715	278	3.390	12.1531

The tensile strength along with the elongation was estimated from the S–S curve and the flexural modulus was tabulated in **Table 3**. From the graph it is understood that the tensile strength of the composite material with 1 *w*/% Inconel content is higher than the normally used GFRP without filler content. The modulus value tends to decrease with an increase in the filler content by 1–5 *w*/%. The modulus value of the GFRP is in the range of 3.8 GPa, and with the addition of 1 *w*/% Inconel filler it increased to 4.8 GPa. This value of the addition of

Inconel filler shows an effective increment of the Young's modulus. The modulus value tends to decrease by 3 *w*/% and 5 *w*/% due to the increased quantity of filler powder dispersed between the fiber and the matrix, thus making a material with increased stiffness. The amount of filler content added to the matrix plays a vital role in deciding the material for a specific application.

Sehajpal et al. discussed how the usage of increased metal fillers in poly-methyl-methacrylate (PMMA) and other resins can reduce the tensile and the flexural properties of the composite materials with the PMMA as a matrix material. In the above case, the epoxy resin is used as the matrix with Inconel as the filler. The flexural properties tend to decrease with an increase in the filler rate, this is because the concentration of the filler affects the flexibility of the material by increasing the toughness of the material.[17] The elastic modulus of about 60 % decreased by 1–3 *w*/% of filler content and about 5 % for 3–5 *w*/% respectively, this effect may be due to the surge in the Inconel particles to the matrix affecting the elastic modulus, the micro grains of the Inconel powder makes the matrix and the fiber to improve the bonding strength by suppressing the elastic modulus. **Figure 3** shows an SEM micrograph of the Inconel filled GFRP composite of the tested specimen. The micrograph clearly shows the improved fiber matrix interaction due to the addition of the Inconel particles. The Inconel particles were closely attached to the fiber matrix interface, enabling the fiber to de-bond from the matrix and improving the delamination strength.

Figure 4 shows the von Mises stress with respect to the pulling force for different Inconel proportions. From the graph it is clear that the GF with epoxy had a pulling force of 1.005 kN, a stress intensity of the GFRP without the addition of fillers is 783 MPa, 1 *w*/% filled Inconel sample showed an increased pulling force of 1.035 kN, the stress intensity on the top and bottom layers was 851 MPa. The fiber volume fraction of the Inconel filled GFRP material showed 64 %. The test was carried out

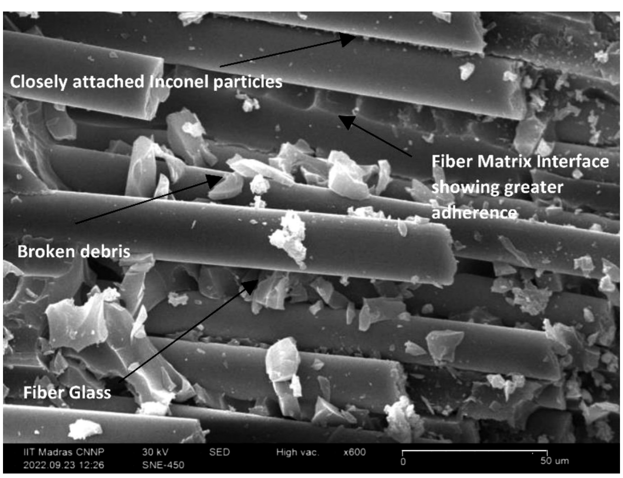

Figure 3: SEM micrograph of the broken GFRP with Inconel 718 filler

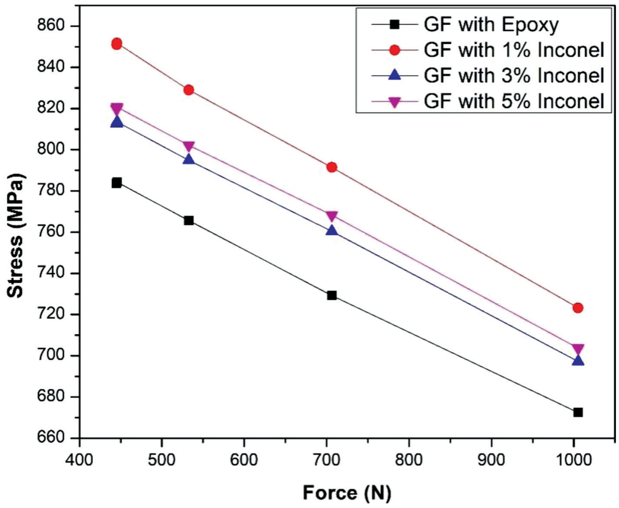

Figure 4: FEA validation results showing the von-Mises stress plot for different Inconel proportions

using the resin-burning-off method. The remaining percentage of 36 % is made up with the epoxy resin and Inconel filler.

The increased stress values were due to the increase in the pull force originating from the ply layers. Similarly, the deformation and the pulling force values were identical for the next series of proportions. The stress values were found to be reduced for the 3 *w*/% and 5 *w*/% Inconel content, it has been clearly understood from the **Figure 4**, that the increased Inconel content reduced the stress values and the bonding strength between the fiber and the matrix. A ply of suitable thickness as per the standard is oriented in the Ansys ACP, 6 layer of thickness of 3 mm with 0.5 mm individual ply thickness was layered one over the other with the fiber and epoxy-resin matrix. The contours from the finite-element method showed the von Mises stress without the addition of filler experiencing higher stress at the bend region on the middle region of the ply, the shear stress at the bending region was recorded as 93.74 MPa in the peeling direction (YZ-orientation). The correlation between the shear stress, deflection and von Mises stress resembles the delamination strength has increased with the addition of the Inconel filler. The results showed a reduction of 0.15 MPa in the shear stress, making the specimen moderately brittle with the addition of the Inconel powder. The water-absorption characteristics of the Inconel filled GFRP composites was identified using the sessile drop method, with the contact-angle values shown in **Figure 5**. The contact angle with the Inconel-filled composites showed a steadily improving curve. The results showed a reduced value for the 0 *w*/% and 1 *w*/% Inconel with hydrophilic characteristics and 3 *w*/% and 5 *w*/% Inconel with hydrophobic characteristics.

The addition of the Inconel filler improved the water-absorption characteristics by 11–12 % from the original value. The flammability test was considered as the important material testing for analyzing the fire-retardant

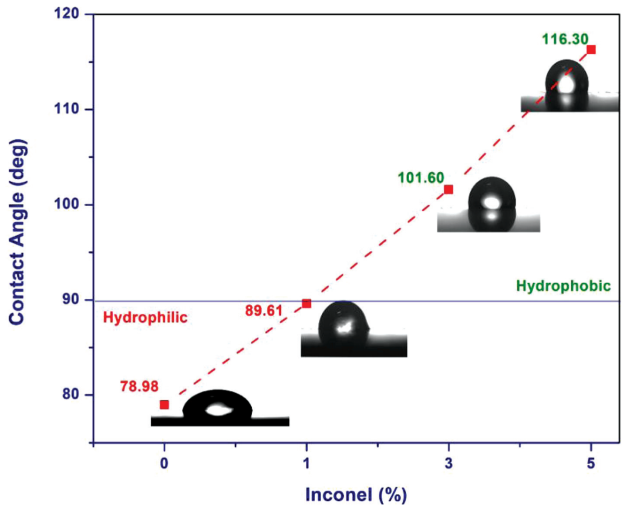

Figure 5: Contact-angle dependence on different percentages for the Inconel 718 filled GFRP composites

characteristics of the composite material. The current flammability test ensures the effect of the addition of the Inconel filler for different proportions. **Figure 6** shows the linear burn rate in the horizontal orientation in mm/sec for the unfilled and the filled Inconel GFRP composites. From the figure it is clear that the rate of burning reduced from 35.4 mm/s to 10.80 mm/s, nearly 69 % of the flame-retardant characteristics improved with the addition of the Inconel filler.

The requirement of the composite materials in various automotive domains has a serious impact on the thermal and flammability characteristics. In order to ensure better flame-retardant characteristics the UL-94 standard was prescribed for detailing the end requirement of the materials. The burn rate was measured in terms of the formula derived with the time and damaged length of the specimen. The linear burning rate is calculated using the Equation (3).

$$V = 60(L/t) \tag{3}$$

where V is the linear burning rate in mm/minute, L is the damaged length in mm, t is the time in s. As per the formula the linear burning rate was calculated and the vertical burn rate was tabulated in **Table 4**.

From the burn-rate results, the vertical burn rate was on the scale of V-2 with a burn time of 251–255 s, with no single drop to ignite the cotton.

4 CONCLUSIONS

The material characteristics and the mechanical properties of the Inconel 718 filled GFRP composites were successfully investigated with a series of mechanical validations and testing. The tensile and the elastic modulus results of the samples with Inconel of 1% showed better results when compared with the unfilled GFRP. The FEA validated results showed that the shear stress was reduced in the YZ orientation along the peeling direction,

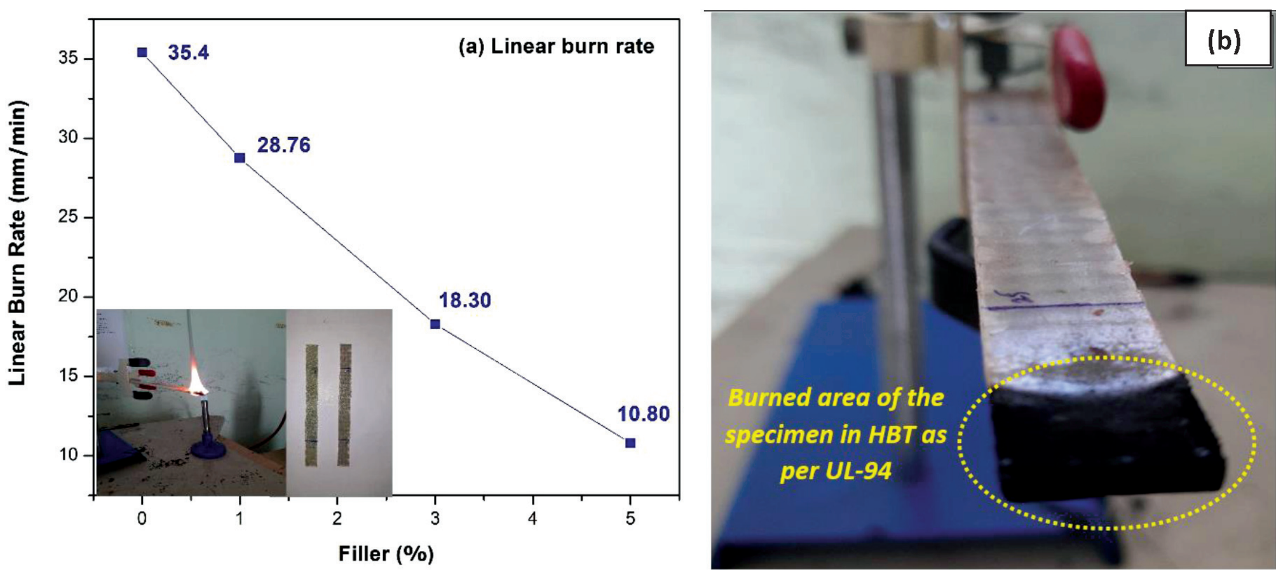

Figure 6: Inconel-filled GFRP composites: a) linear burn rate, b) horizontal burn-tested specimen

Table 4: Burn rate of the GFRP with epoxy and (1, 3 and 5) *w*/% of Inconel 718 filler

Sample No.	Inconel 718 (*w*/%)	Damaged length (mm)	Time (s)	Linear burning rate (mm/sec)	Combustion level	Laminates self-extinguished after flame removal
1	0	25	59	35.4	V-2	NO
2	1	35	73	28.76	V-2	NO
3	3	15	50	18.30	V-2	NO
4	5	13	72	10.80	V-2	NO

which enhanced the bonding strength between the fiber and the matrix with Inconel 718 filler. It was observed that the material had a brittle nature with the addition of the filler. The delamination strength and the shear stress were found to be higher for the 1 *w*/% Inconel 718 content. The contact-angle analysis of the unfilled and filled GFRP showed higher values for the 5 *w*/% Inconel 718 filled material compared to the 1 *w*/% and 3 *w*/% , the contact angle of 5 *w*/% filler was 116.30°, the incorporation of the Inconel 718 alloy with the epoxy matrix has the same characteristics as the metal oxides. The hydrophobic characteristics were improved with the addition of the metal alloys with the epoxy coatings, thus enabling the material to behave as hydrophobic in nature. The flammability characteristics of the 5 *w*/% Inconel 718 filled material showed reduced linear burn rates of 10.80 mm/min. The VBT showed V-2 inference as the flame is not self-extinguishing up to 251–255 s. The increased VBT range is due to the fiber and the matrix cross-orientation causing the material to burn with a little smoke. Thus, the overall results showed an increase in the Inconel 718 material enhanced the mechanical and flammability characteristics of the GFRP composite laminates.

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