An experimental study of De-Icing property using Reduced Graphene Oxide - Boron Nitride Nano Powder in CFRP

Akash Raja A\textsuperscript{1}, M S Nisha\textsuperscript{2}, J Jatin\textsuperscript{1}, Joseph Noel Kiren\textsuperscript{1}

\textsuperscript{1} Bachelor of Technology, School of Aeronautical Science, Hindustan Institute of Technology and Science, Chennai, Tamil Nadu 603103, India.
\textsuperscript{2} Assistant Professor, School of Aeronautical Science, Hindustan Institute of Technology and Science, Chennai, Tamil Nadu 603103, India.

Abstract. Icing is one of the critical factors that affects the flight of the aircraft in ways of drag. In this study, the rate of de-icing was experimented with the composite material using Boron Nitride Nano Powder (BNNP) and Reduced Graphene Oxide (rGO). Boron Nitride are highly thermal conductive and lesser toxic, and Graphene allows to be a good conductor of heat and electricity and thereby, these nanomaterials are used as a filler for de-icing application. The filler is mixed in Epoxy Resin using ultrasonicator. In order to get better specimen surface of composite, ultrasonic mixing method is used instead of hand-mixing. To fabricate Carbon Fiber Reinforced Polymer (CFRP) Hand Lay-up Method is carried out. The material characterization is done by X-ray Diffraction (XRD), and Scanning Electron Microscope (SEM). Mechanical properties are studied with Dynamic Mechanical Analysis (DMA), Thermo-Gravimetric Analysis (TGA), and Water Droplet test. Moreover, the Carbon Fiber Reinforced Polymer surface shows extraordinary de-icing effect contrasted with the non-functionalized surface. This study has proved that the de-icing property is very much effective with Carbon Fiber Reinforced Polymer where Graphene and Boron Nitride as the filler.

1. Introduction

It is well known that cold weather can be the scourge of air travel if you've ever had a flight delayed by a snow-covered runway. Far from the ground, though, ice has even more serious repercussions. Super-cooled water droplets may create layers of ice on aerodynamic surfaces like wings, propellers, or jet intakes as an aircraft flies through clouds at sub-zero temperatures. This can increase drag and dramatically decrease lift, making it hard to fly the aircraft [1]. De-icing is a technique to remove or melt ice, snow, or frost formed on the surface of an aircraft. Icing is one of the major problems on an aircraft as it obstructs the flight of the airplane, drag is increased, and lift is also disturbed. It is a serious complication on an airplane which can cause severe reparations like structural damages as the ice breaks and get ingested into the engine. Ice formed on the rotor blades and at jet intakes may result in flaming of the engine. This could lead to major crash which can injure all the passengers on the airplane. For reference, as indicated by ongoing FAA studies, airplane crashes because of icing took around 30 lives, wound 14 others, and result in $96 million in property damage yearly in the United States (McGehan, 2002). Cancellations and delays because of cold climate can cost airliners a huge number of loss in a solitary day. On March 20, 2000, icing conditions at Denver International Airport constrained Air Wisconsin to drop 152 flights. The US dropped 159 outbound and 140 inbound flights the very day, for the most part on account of climate ("Airlines Get New Tools to Avoid In-Flight Icing," 2002). As David Hinson, then FAA administrator, clarified the company’s situation: "Technology has not advanced to the point of providing a reliable means to assess in-flight icing conditions with that degree of accuracy or specificity" (Cole, 1997, B 1) [2,3].
There are various methods for de-icing such as shovelling, conductivity, infrared beam, and chemical fluids. There are methods of de-icing while flight such as pneumatic boots, weeping wings, bleed air heating surfaces, electro-mechanical. All the de-icing methods have their own weaknesses like for shovelling, it cannot be done all the time and for every part of the aircraft. Spraying of chemical fluids has its own hindrances— the amount of money spending on these chemicals are huge that can create the airline company a definite loss too. It also takes time to deice which can delay the departure. Electric heating surface is very efficient and also have less percent of drawbacks comparatively [4,5].

In this research, Carbon fibres, graphene and boron have been used. These components are basically very good conductors of electricity where for even giving a less input of electric current, the surface gets heated [6,7]. This benefits the rate of de-icing property. Graphene is one among the versatile substances that can be occupied or used for many applications. Its properties are very much effective for deice application where the thermal conductivity is high, and additionally, it is corrosion resistance. The heat transfer is finest in the composite where it is coated with graphene [8]. Similarly, graphene oxide also depicts good thermal conductivity, heat transfer [9]. Bhatti et al. [10] inspected the Electromagnetohydrodynamic (EMHD) flow with heat transfer on 3rd grade fluid covering minute particles or atoms. In relation to subjects of heat transfer, Ellahi et al. [11] formed a prototype to deliberate the divergent questions. The above references deliver theoretical and numerical explanation to heat transfer.

In the study, composites are reinforced with Boron Nitride Nano-powder which are also a good thermal conductor that transfers heat and have slightly lesser properties than that of graphene [12].

2. Experimental

2.1. Material Selection

To experiment the various tests, the following materials are selected for the fabrication of the composite. Carbon Fiber Reinforced Polymer sheets, Araldite LY 556, Hardener HY 951, rGo, BNNP (fig 1,2).

![Fig 1. Araldite LY 556, Hardener 951, Boron Nitride Nano Powder, reduced Graphene Oxide](image1)

2.2. Fabrication of Composites

2.2.1 Synthesis of Carbon Epoxy Composite with rGO

Hand Layup process is brought out to form the carbon epoxy composite. Hand Layup is one of the traditional and still used molding process that provides the composite to be durable and flexible. This method proceeds by physically setting down individual layers or ‘plies’ of a type of reinforcement called as ‘prepreg’. Hand Layup method has a low-cost setup and is very efficient, and is also adaptable to any
new changes made to the design [13]. This process is executed by pressing on the CFRP with the resin and hardener as the filler. First, to make sure the surrounding has no oil or granules of dirt, thinner is used to remove it. Here, 15x15 cm is taken for a CFRP layer where it is placed on a transparent film greased with wax for not sticking onto the surface. Araldite LY 556 of 102.827 g along with 5% weight of epoxy to be of rGO are mixed and dispersed using an ultrasonicator. After a period of 15 minutes, hardener HY 951 of weight 10:1 of resin is stirred well, and used as a filler in the epoxy composite. Each layer is coated with the mixture using a brush and a roller and another layer of carbon fiber is kept above. This step is repeated until the sufficient thickness is obtained, here it is 2 mm. Later it is pressed and left for curing for roughly 36-48 hours. The fabricated composite is attained with no air gaps or voids with the help of Hand Layup technique.

2.2.2. Weight Percentage of components in the Composites

The above process is used for other two fabrication of specimens where BNNP, and combination of rGO and BNNP are present. The table below shows the percent and weight of rGo and BNNP in each and every sample for the study.

| Sample No. | Sample name     | Epoxy Resin | rGO  | BNNP |
|------------|-----------------|-------------|------|------|
| 1          | rGo [100%]      | 102.827     | 5.14 | -    |
| 2          | BNNP [100%]     | 102.827     | -    | 5.14 |
| 3          | rGO[75%] + BNNP[25%] | 101.32     | 3.85 | 1.285 |

2.3. Characterization Techniques

i. **X-ray Diffraction:**
X-ray diffraction studies of CFRP is carried out to study the CFRP compositions, rGO and BNNP nanoparticles structure incorporated, using Empyrean XRD [14-15].

ii. **Scanning Electron Microscope:**
SEM is used for imaging the microstructure and morphology of the rGO & BNNP nanoparticles in the CFRP, using TESCA VEGA [vacuum: 10^-6, accelerating voltage:30KV & sputtering surface: gold] [15-16].

iii. **Dynamic mechanical analysis:**
Using DMS6100 DMA machine (SII nanotechnology, Japan) we find the mechanical properties [storage modulus, loss modulus etc] of the CFRP samples [17-18].

iv. **Thermo-Gravimetric analysis:**
Using TGA/DSC 3+ machine (Mettler Toledo, Switzerland) we determine the thermal performance and stability of the CFRP samples fortified with rGO&BNNP at nitrogen atmosphere, between a temperature range of 30-600°C & at a rate of 10°C/min [19-20].

v. **Water Droplet Testing:**
To prove and test the de-icing capabilities, a water droplet test was carried out, water droplets were scattered on the CFRP sample surface and cooled to about -15°C, until it was frozen (and returned to non-sub-zero cool temperature), and a small current from a 9V battery was passed through it with help of clamps for period of time until the de-icing was achieved and the frozen droplets detach from the surface, this happens at a faster rate (i.e. shorter time) for rGO dominant CFRP due to its superior electrical and thermal properties.
3. Results and discussion
To determine and analyze the characteristics of the materials or samples, the following tests are carried out.

3.1. Scanning Electron Microscopy (SEM):

Scanning electron microscope measurement were carried out to study the topography and surface morphology of the reduced Graphene oxide and Boron Nitride Nanoparticles as a filler in the modified CFRP laminate. Fig 3. a, b, c represents the SEM images taken in different magnification. The image shows the high wrinkled textured surface and it seems like a thin layered structure of rGO which covers large area [21]. The BNNP were visible in the images which shows the predominantly irregular flake shape. The predominates shape was nearly hexagonal disk particles [22].

Fig 3. (a) SEM image of CFRP composite (75%rGO and 25% BNNP) at magnification of 20 µm; (b) SEM image of same composite at magnification of 5 µm; (c) SEM image of the same composite at total magnification of 1 µm.

Fig 3.c. shows the SEM image of high resolution where the BNNP are in 60-90 nm which were used as reinforcement fillers and the nanocomposites reinforced in different weight percent of 75% rGO.
and 25% BNNP of such nanoparticles under ultrasonic mixtures. The fillers are expected to modify the structural and mechanical properties in terms of De-icing.

### 3.2. X-Ray Diffraction (XRD) analysis:

**Fig 4. (a) XRD graph of 100% rGO sample; (b) XRD graph of 100% BNNP sample; (c) XRD graph of combination of 75% rGO and 25% BNNP**

Fig 4. a, b, c displays the XRD patterns of rGO, BNNP, and the combination of both. In Fig 4.c, it shows a strong and sharp diffraction peak at 2θ approximately equal to 27° but the d-spacing is very much narrow. Fig 4.a has broad and weak diffraction peak comparatively and it has moved at angle 2θ approximately equal to 24°-25° but has greatest intensity. This is owing to on the basis of the arrangement of layers than with BNNP [23].

### 3.3. Dynamic Mechanical Analysis (DMA):

For the composite having 100% rGO content, the Fig 5. a, b, c represents the storage modulus, loss modulus, and damping peak respectively. Fig 5.a denotes the storage modulus where it is higher for higher frequency. Since the values of E’ drops when temperature increases, the drop occurs around 80 °C. The modulus’ values swiftly decrease until it reached the temperature of 100 °C, and it is unaffected after that [24].
Fig 5.a. The storage modulus of 100% rGO content at different frequencies

In fig 5.b., the values of loss modulus are depicted where it tops at temperature 80-100 °C for almost all the frequencies, and then decreases. This shows that there is a loss in the form of dissipation of heat at this range of temperature [25].

Fig 5.b. The loss modulus of 100% rGO content at different frequencies

The ratio between loss and storage modulus is known as the damping factor [26]. In fig 5.c., for lesser frequency, the tan δ value is higher because the ratio is greater in lower frequencies.

Fig 5.c. The tan δ curve for 100% rGO at different frequencies

In the same way, for other specimens, the discussions and analysis for each graph are similar. Fig 6 a, b, c represents the composite containing 100% BNNP.
Fig 6. (a) Storage modulus values for 100% BNNP at different frequencies; (b) Loss modulus values for 100% BNNP at different frequencies; (c) The tan δ curve for 100% BNNP at different frequencies.

For composite having 75% rGO and 25% BNNP, Fig 7. a, b, c characterises its dynamic mechanical properties.
Fig 7. (a) Storage modulus values for 75% rGO and 25% BNNP at different frequencies; (b) Loss modulus values for 75% rGO and 25% BNNP at different frequencies; (c) The tan δ curve for 75% rGO and 25% BNNP at different frequencies.

3.4. Thermo-Gravimetric Analysis:

From Fig 8, it can be noted that CFRP fortified with nanoparticle hardeners like rGO and BNNP have a 3 step degradation curve with temperature increase: a negligible to almost no initial degradation occurs from 0-300°C accounting only to about 0-2% weight loss, An adverse and exponential increase in degradation occurs between 300-400°C which contributes to the majority weight loss at about 75% of all weight loss and 30% of the whole initial sample weight, The steep loss curve then eventually flattens at around 400°C and degrades at a relatively steady rate to the final limit temperature of about 600°C accounting for about 5-15% of weight loss depending on the sample[23].

3.5. Water droplet test:

In this test, the three composites are connected to the battery each. The electricity is passed and the timings are noted for each until the frozen water droplet runs over. The sample containing 100% rGO as the filler in weight percentage terms give 40.05 seconds as the rate of de-icing. For
sample containing filler as 100% BNNP, the time for de-icing is 53.63 seconds, and sample containing fillers with 75% rGO and 25% BNNP gives the rate of 46.62 seconds. Fig 9. shows the position of the composite along with the attachments for the technique.

Fig 9. Water droplet test with a single composite attached to a 9V battery

4. Conclusion

This experimental study investigates the characteristics of the composite polymers containing rGO, BNNP, and both in the epoxy matrix. The laminate of CFRP with rGO and BNNP fillers at different weight percentage was manufactured by Hand-Layup technique. The presence of rGO and BNNP in the epoxy matrix was proved by XRD analysis. The microscopy results of the fabricated CFRP also confirmed the presence of rGO and BNNP, and the SEM image shows more intimate contact and more interaction between the carbon fiber and epoxy with fillers, although surprisingly good adhesion was observed in the CFRP composite. The thermal stability of the composite increased with the increasing content of rGO [27] and with a more significant improvement in the fillers with pure rGO which was determined by TGA. The DMA results clearly show that the damping factor is less in sample containing filler weight percentage of 100% of rGO comparing with the other two samples, and according to the water droplet test, specimen with filler of 100% rGO shows faster rate of de-icing.

Thus, with respect to all the norms of testing methods, composite containing 100% rGO as filler have the superior behaviour and therefore, the de-icing property and the mechanical properties of this particular composite are favoured highly.

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