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MECHANICAL PROPERTIES EVALUATION OF CARBON PHENOLIC COMPONENTS WITH NANO ZrO_2 AND GRAPHENE OXIDE FILLERS

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Abstract: The effects of hybridization and the stacking sequence of hybrid composite materials under pressure-based impacts were investigated. Work focused on mechanical properties were optimized. 1.5% addition with 20 bar and curing temperature of 90°C given better results in mechanical properties compared with other samples in previous experiments done. Further work carried out with parameters with curing temperatures 50, 70 and 90°C and pressure 15, 20, 25 bar with the addition for each sample a standard amount of 1% graphene oxide added in fabrication process. The injection takes from the top nozzle middle nozzles and diagonal left nozzles for the following experimentation work. The optimal samples were tested for compressive and 3 point bending tests. L9 Taguchi method of DOE selected for the experimentation. The mechanical properties evaluation of carbon phenol components enhanced with Nano ZrO_2 and graphene oxide fillers showed significant improvements in both strength and durability. These fillers contributed to better thermal stability and increased resistance to wear and tear.

Key words: Carbon/Phenol, Laminates, vacuum impingement method, ZrO_2 , Grapheme, Nano Fillers.

1. Introduction

Carbon fibre/phenolic resin (CF/PR) composites have been extensively employed as high temperature structures in aerospace and space industries. Phenolic resin is a crucial component of the composite, as it can form a char layer that is capable of withstanding high

temperatures, thereby protecting the main structure from destruction or degradation. With significantly better ablative properties than other polymer-based composites, CF/PR composites are prime candidates for shielding structures from the harsh thermal and aerodynamic conditions. Srikanth *et al.* [1] manufactured the high

ablation resistance rayon-based carbon-fabric/Ph composites using nano-SiO₂ powder as a filler material. Nano-silica-filled composite exhibited higher ablation resistance when compared to the unfilled composite. Asaro *et al.* [2] used mesoporous SiO₂ particles as reinforcement of a Ph resin to develop new ablative materials. They concluded that the composite with 20% silica particles achieved the best performance because the mesoporous structure was responsible for the improved ablation performance. Duan *et al.* [3] studied the effects of borosilicate glass and polycarbosilane on the oxidation and ablation behaviors of carbon fiber/Ph resin composites. They found that the fillers changed the oxidation mechanism of the composite at temperatures above 1,000°C. This

2.Literature review

Kwon YJ et al [6]. A grapheme/carbon nanotube hybrid material stabilized in an aqueous medium was coated on carbon fibers by anodic electrophoretic deposition. Chemically oxidized grapheme, grapheme oxide, was used as a stabilizing agent for dispersion of carbon nanotubes and as a transport medium for the grapheme oxide/carbon nanotube hybrid during electrophoretic deposition. L Asaro et al [7] .Mesoporous silica particles and carbon black were selected as fillers for a resol-type Phenolic resin, to be used as a matrix for

modification improved the strength of the composite after oxidation. Wang *et al.* [4] evaluated the effects of zirconium carbide content on thermal stability and ablation properties of carbon/Ph composites. They showed that increasing ZrC content could lead to an evident increase in char yield but an observable reduction in linear ablation rates and back-face temperatures because of the formation of the ZrO₂ layer on the ablation surface. Chen *et al.* [5] introduced ZrB₂ particles into Ph resin and prepared modified carbon-phenolic (C-Ph) composites with this resin as a matrix. ZrB₂ could improve the thermal stability of Ph resin. The formation of ZrO₂ and B₂O₃ could notably improve the ablation resistance of C-Ph composites during the ablation process.

ablative materials. Composites were processed with the modified polymer and carbon fibers were used as continuous reinforcement. The ablative properties of the materials obtained were studied L. Paglia, et al [8] In this work, two different kinds of carbon-Phenolic ablators with a density of 0.3 g/cm³ were manufactured and their mechanical and thermal properties were experimentally evaluated. The thermal protection performances of the developed ablators were assessed in a hypersonic plasma wind tunnel facility, setting representative enthalpy and heat flux

conditions (6 and 13 MW/m²), consistent with atmospheric reentry missions from high energy orbits M Satyanarayana Gupta [9] Carbon-Phenolic composites are meant for heat protection of the aerospace structures like aircraft skins, nozzles and heat shields during the aerodynamics loading conditions. Phenolic resin matrix composites are used as ablative liners to resist thermal and erosive environment. The manufacturing process of Carbon Phenolic composite structure requires curing. Megha Choudhary et al (2022) [10] Since their discovery in 1991, carbon nanotubes (CNTs) have found widespread use in various industries, from aerospace to energy, due to their excellent mechanical, thermal, electrical, and tribological properties. Mohan Kumar (2017) [11] In solid rockets, the propellant are burned in the combustion chamber and the exhaust gases of very high temperature are accelerated through a convergent-divergent nozzle. The chemical energy of the propellant is converted into heat energy which in turn gets converted into kinetic energy while expanding through the convergent-divergent nozzle. Mohan Kumar, L [12] Ablative composites are an elite class of polymer composites used for thermal protection in aerospace applications like solid rocket motor nozzles, liquid propellant engines, nose caps and external surfaces of atmospheric re- entry space vehicles to protect them from the extremely severe operating conditions of high temperature, pressure and particle impingement. Mohan Kumar.L [13] The external surface of a re-entry space vehicle experiences high heat flux and temperatures of the order of 2500°C during re-entry into the atmosphere and the internal contour of a rocket motor nozzle has to encounter temperatures above 2000°C during its operation. Special Thermal Protection Systems (TPS) are employed to protect the rocket hardware and re-entry capsule from such extreme thermal environments. Raghu Raja P et al (2022) [14] The effect of Nano zirconia, Nano titanic and fumed silica on the mechanical, thermal, and ablation behavior of carbon-Phenolic (C-PH) composites is investigated. e inorganic Nanos fillers at different loading percentage are used to prepare Nano-C-PH. panels by the compression molding technique. Dispersion of Nano. Callers are conformed through SEM analysis. Ren, Yu; Lin et al [15] Boron-modified high-orthodox Phenolic resins (BPRs) were prepared under normal pressure by using Phenol and formaldehyde as raw materials, zinc acetate, and oxalic acid as catalysts, and boric acid as a modifier. Boron- modified Phenolic fibers (BPFs) were prepared by melt spinning and curing in a mixture of formaldehyde and hydrochloric acid, followed by a heat treatment under high temperature. S, SUBHA; Singh [16] The present experiments have been conducted to

investigate the thermal, ablation and formulation based on previous literature mechanical properties of and fabrication of mono and hybrid Carbon/Phenolic(C-PH) composites loaded composites Mixing ratio of epoxy and the with Zirconia (Zr) coated grapheme Nano hardener is 100:25 by weight. After fixing platelets (Gnp) hybrid filler. The Zr/Gnp the reinforcement, filler and epoxy wt.%, hybrid filler was synthesized by sol-gel hand lay-up process is implemented for technique and C-PH composites were stacking the carbon fabric with proper fabricated using hand layup method. phenol coating. A 300x300X1.5mm mould

3. Methodology

Fabrication of composites started with surface modification of the resin and filler. A solution of silane coupling agent and acetic acid was made by mixing in 1:5 weight ratios respectively. Later ZrO_2 is mixed in prepared solution in 1:1 proportion by weight using ultrasonication process for 30 min. After that, ZrO_2 particles were sieved and dried at 110 °C for 3 h. Figure 1 illustrates surface treatment of ZrO_2 particles. Final

has been used to prepare the laminates with carbon fibre added with ZrO_2 fillers. Taguchi method of DOE with 3 levels and three parameters with L9 orthogonal array considered for experimentation and optimized the parameters using Mini-Tab software. SEM analysis carried out for the best samples to check the filling of ZrO_2 affect on mechanical properties of Tensile and Flexural strengths. Any composite is synthesized from a reinforcement fibre and a resin matrix.

3.1 Carbon Fiber: Carbon fibers possess strength due to the relatively high melting low density, excellent tensile and point. compressive strength, and high char

Table:1 Properties of as-received carbon fiber as procured details

Type of fiber	Density (g/cm ³)	Coefficient of thermal expansion (x10-6 /°C)	Thermal conductivity (W/m.K)	Tensile strength (GPa)	Young modulus (GPa)
carbon fiber (rayon-based)	1.8-1.9	0.4-0.75	20-80	3.4-6.2	220-450

3.2 Phenolic Resin: In current study, the resistance and high char yield compare to phenolic resin was used as a matrix phase other polymeric matrix due to its good ablation resistance, thermal

Table:2 Properties of as-received phenolic resin

Type of resin	Density (g/ml)	Thermal Conductivity (w/mK)	Viscosity (cp) @300 °C
Phenolic Resin	1.32	0.25	150-350

3.3 Zirconium Dioxide: Most studied ceramics among the UHTC materials is zirconium dioxide. At room temperature the zirconia, be -have as a monoclinic phase structure and transitions to tetragonal and cubic phase structure when treated to some high temperature

Table 3: Properties of synthesized ZrO₂

Filler	Density (g/m ³)	Thermal Conductivity (w/mK)	Melting temperature (°C)	size (μm)
Phenolic Resin	5.65	1.7	2700	5

3.4 Graphene oxide:

It is used in nanocomposite materials, as a surfactant with some overlaps between polymer composite materials, energy storage, these fields. biomedical applications, and catalysis, and

Table 4: Properties of synthesized Graphene oxide

Filler	Density (g/m ³)	Thermal Conductivity (W/m·K)	Melting temperature (°C)	size (μm)
Graphene oxide	2.267	~5000	4510	4

The reinforcement is the main load bearing ablative composites, the most commonly member in the composite which gives the used reinforcements are carbon and silica strength, stiffness and structure while the fibers due to their high temperature resin matrix serves to transfer the load durability. Hence, these were chosen as uniformly and binds the fibres together. For reinforcement for this study. Resol-type

phenolic resin was selected as the matrix due to its high char yield and char strength. Phenolic resin is synthesized by the condensation reaction of phenol and formaldehyde. The resin was procured from a standard supplier and was characterized for all the important properties of the matrix. For each samples a standard amount of 1% graphene oxide added in fabrication process.

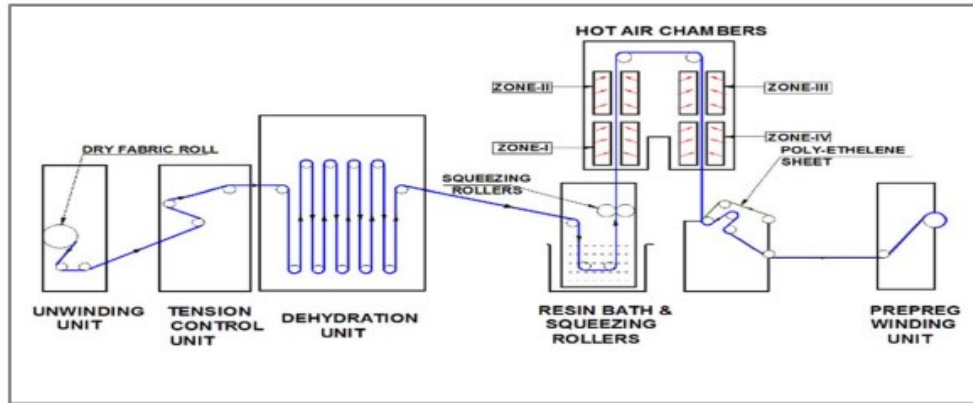


Figure 1: Vacuum impregnation process of laminates

Resol-type phenolic resin was prepared until the pH reached a value of 6.8–7.0. The using a formaldehyde to phenol molar ratio resol was dehydrated in a rotary evaporator equal to 1.3 under basic conditions, as by vacuum at 70°C. During this step, follows. Phenol and formaldehyde water viscosity was measured every 15 min to solution (37 wt%) was placed in a 1-l avoid an excess increase in this property stainless steel reactor with a low-velocity which could generate problems in the stirrer, a thermometer, and a reflux condenser. processability of the materials. The resin The pH was kept at 9.0 with a 40 wt% obtained was kept at 10 degree centigrade solution of sodium hydroxide (NaOH) and until it was used. The phenol and the mixture was allowed to react for 2 h at formaldehyde used for the synthesis added 90°C. After that, the mixture was neutralized with ZrO₂ and Grapheme oxide sonicated with a 7 wt% solution of boric acid (HBO) Nano fillers.

Samples for testing



Figure 2: Carbon fibre laminate with and without phenol coat



After completing vacuum infusion 695 and 790. Samples prepared @ 0, two types of samples prepared at thickness 22.5 and 45. Tested on the 2-point bending of 1.5mm and 3mm and at tensile testing machine ADT- temperatures 00Cto900C. samples prepared 4000KN, as per the ASTM standards D-3039,



Figure3.shows the samples preparation 1.5mm thickness



Figure 4 Shows the samples preparation 3mm thickness after phenol coat

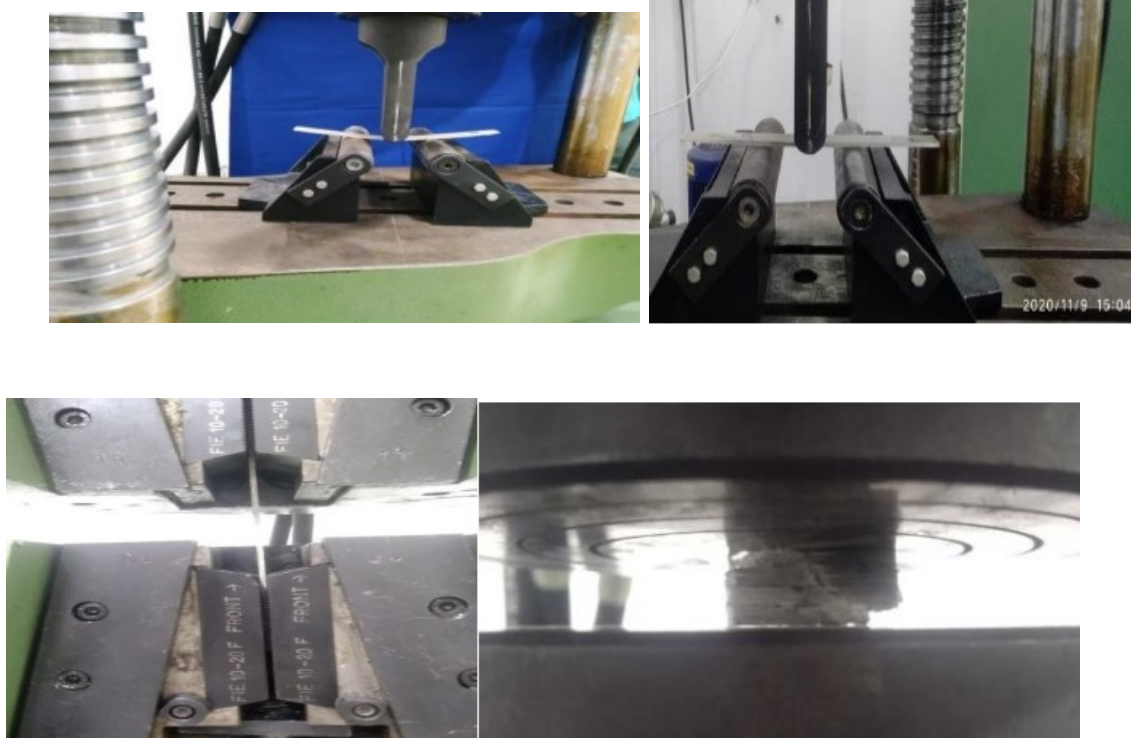


Figure:5 shows the testing procedures for UTS, flexural, Impact, compression

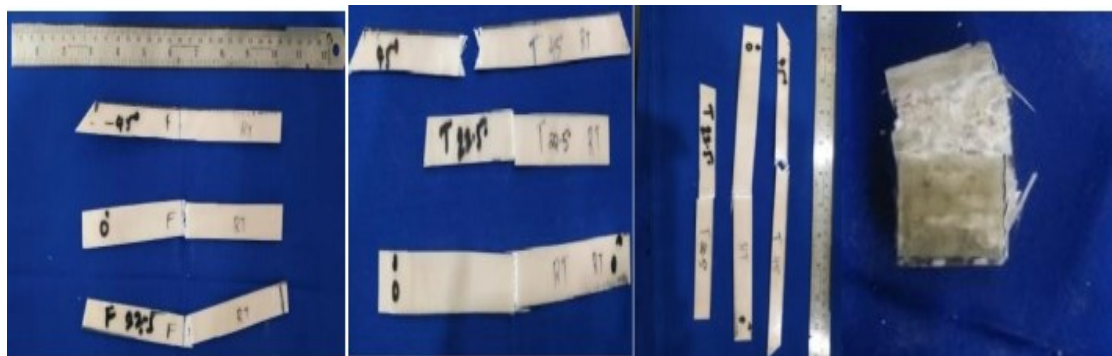


Figure :6 shows the broken samples of UTS, flexural, Impact

4. Results and Discussions

various mechanical tests to evaluate their strength, flexibility, and durability. The following are the results after testing the laminates. Graphene oxide combined with ZrO₂ Nano filler to create carbon phenol material as shown in the tables 5 and table6. These laminates were subjected to

Table:5 Experiment results of tested samples with 1.5 and 3 mm thickness

S.NO	Filler content (w%)	Pressure (bar)	Curing temperatures ($^{\circ}\text{C}$)	1.5 mm top		3mm top nozzle	
				Tensile strength (Mpa)	Flexural strength (Mpa)	TS	FS
1	1	15	50	150.4	96.6	195.52	121.716
2	1	20	70	154.6	97.5	200.98	122.85
3	1	25	90	169.1	104.2	219.83	131.292
4	1.5	15	70	178.3	109.9	231.79	138.474
5	1.5	20	90	183.6	119.4	238.68	150.444
6	1.5	25	50	181.1	116.2	235.43	146.412
7	2	15	90	179.2	111.3	232.96	140.238
8	2	20	50	178.8	107.9	232.44	135.954
9	2	25	70	168.7	102.1	219.31	128.646

Table:6 Results for injecting directions from front nozzles

	1.5mm front		3mm front		1.5mm left		3mm left	
S.no	TS	FS	TS	FS	TS	FS	TS	FS
1	162.432	98.339	207.251	125.367	165.031	106.206	213.469	112.578
2	166.968	99.255	213.039	126.536	169.639	107.195	219.430	113.627
3	182.628	106.076	233.020	135.231	185.550	114.562	240.010	121.435
4	192.564	111.878	245.697	142.628	195.645	120.828	253.068	128.078
5	198.288	121.549	253.001	154.957	201.461	131.273	260.591	139.150
6	195.588	118.292	249.556	150.804	198.717	127.755	257.042	135.420
7	193.536	113.303	246.938	144.445	196.633	122.368	254.346	129.710
8	193.104	109.842	246.386	140.033	196.194	118.630	253.778	125.747
9	182.196	103.938	232.469	132.505	185.111	112.253	239.443	118.988

Table:12 Taguchi Prediction and rankings for the above laminates

	Parameters			Taguchi prediction				Rank
	A	B	C	S/N Ratio	Mean	St Dev	Ln (St Dev)	
1	1	1	1	43.108	166.538	74.333	4.305	8
2	1	2	2	43.020	165.817	75.620	4.324	9
3	1	3	3	43.642	177.920	80.047	4.382	7
4	2	1	2	44.104	187.771	84.583	4.435	6
5	2	2	3	44.924	203.385	88.864	4.490	1
6	2	3	1	44.511	195.519	86.806	4.464	3
7	3	1	3	44.197	191.316	88.937	4.488	2
8	3	2	1	43.982	186.960	86.733	4.459	4
9	3	3	2	43.696	182.730	88.167	4.482	5

Table:8 Compressive Strength for optimal best 4

S.NO	Temperature ° C	Thickness	0°	22.5°	45°
1	90	1.5	173.40	168.55	170.10
2	90	3	153.40	149.30	145.90
3	90	1.5	163.55	172.40	167.55
4	90	3	147.30	143.40	142.55

Compressive strength given notable results not much difference in angle of sample taken, the sample in 1.5 thickness given good result. Compressive strength decreased with the increase of thickness and low in temperature value. Almost similar strengths attained in 3mm thickness samples, clear variation of more than 10%, 1.5 thickness samples attained than 3mm.

Table:9 Shear Strength

S.NO	Temperature ° C	Thickness	0°	22.5°	45°
1	90	1.5	60.15	74.88	109.23
2	90	3	57.53	70.55	101.23
3	90	1.5	61.59	71.57	110.57
4	90	3	60.30	69.55	104.59

Shear strength also proportioned to flexural but the outmost strength decreased, strength increased with angle of sample taken, only 3mm at 45 degree given variant result, rest of others are normal proportionate in strength deviation.

5. Conclusions:

The results indicated that the addition of ZrO₂ and graphene significantly enhanced the mechanical properties of the carbon phenol laminates. The improved performance was attributed to better dispersion and strong interfacial bonding between the matrix and the added nanoparticles. These improvements suggest potential applications in advanced composite materials where increased strength and durability are required. Improved thermal stability and increased tensile strength were observed, suggesting potential for advanced composite applications.

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