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Investigating the effect of carbon fabric grammage on the mechanical and erosion properties of CFRP reinforced with ZrB_2/SiC ceramic particles at a temperature of 3000°C

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Abstract
This research investigates the mechanical performance, ablation and oxidation of insulation based on carbon-epoxy Novalac reinforced with ZrB_2/SiC ceramic particles with different grammage of fabric. The purpose of this research is to investigate the effect of carbon fabric grammage on epoxy novalac carbon insulation reinforced with ZrB2/SiC ceramic particles. Three samples including Novalac epoxy resin base with 60% SiC + 40% ZrB_2 and 120, 200 and 600 gr/m^2 of carbon fabric were made using hot press. The mechanical and thermal properties of insulation samples with different grammage using Oxyacetylene, bending and tensile tests were evaluated. To investigate the surface morphology, X-ray energy distribution elemental analysis (EDS) and SEM scanning electron microscope and X-ray diffraction (XRD) phase analysis have been used to identify the existing phases. The results showed that the mechanical properties and erosion resistance of the composite improved by increasing the grammage of the fabric. The biggest weight reduction and thickness reduction were in the insulation samples with 600 gr/m^2 (G3) and 120 gr/m^2 fabric (G1), respectively. Also, the G3 sample had the lowest temperature behind the insulation, below 150 degrees Celsius and had the highest mechanical properties, with 486.35 MPa of tensile strength and 677.18 MPa of bending strength. Finally, morphological studies showed that the presence of ZrB2/SiC particles leads to the creation of refractory phases such as ZrO_2, SiO_2 and $ZrSIO_4$, which increase the resistance to hot erosion of carbon epoxy Novalac composites.

Keywords: Composites, ablation, additive.

1. Introduction

Recent research has focused on improving ablative properties through matrix modification with ceramic particles and varying fabric weights. For instance, Paiva et al. noted that F584/PW laminates exhibited the highest tensile strength [1]. Parvin et al. showed that microwave-cured carbon fiber epoxy composites had a 9% increase in interlaminar shear strength [2]. Similarly, Irum Rafiq et al. found that varying fabric weights improved resistance to mechanical stress and environmental conditions [3]. Farhana confirmed these improvements in SC-15 epoxy reinforced with carbon Nano fibers [4]. Additional studies include Kim et al. on thermal conductivity in epoxy composites with alumina and aluminum nitride fillers [5], and Rahmani et al. on the mechanical performance of multilayer epoxy/carbon fiber composites, identifying EM500 epoxy resin as superior [6]. Liu et al. studied the ablative resistance of C/C-SiC-ZrB2 composites exposed to oxyacetylene, highlighting the formation of a protective oxide layer [7]. While there are many studies on the advantages of ceramic particles in epoxy-based insulations, no comprehensive research has examined the effects of individual components on the mechanical and ablative properties of carbon insulation with varying fabric weights using Novolac epoxy resin reinforced with ZrB2/SiC particles.

2. Materials

In this study, Novolac epoxy resin (EPN1179 from Huntsman), DICY curing agent, and Diuran accelerator (from Sigma Aldrich) were used as the composite matrix. MEK solvent was added to reduce the viscosity during manufacturing. Silicon carbide particles (15 microns, Ovonic, Germany), zirconium diboride particles (18 microns, Merck), and plane-woven carbon fibers (120, 200, and 600 g/m², T300, Yancheng, Jiangu, China) based on PAN were used. Microstructure and morphological changes of the composites were examined using a SEM X-MAX 50 (Oxford, UK), while phase analysis was conducted using a D8 ADVANCE x-ray diffraction machine (Bruker Elman, EDS).

3. Manufacturing and Testing

The preparation of the resin began by heating 80g of it at 80°C for two hours to convert it from a semi-solid to a liquid state. To maintain this state, a solvent (MEK) was added, adjusting the viscosity to 20% by weight. The resin was stirred at 60°C and 500 rpm on a magnetic stirrer. Curing agents (Dicy and Diuran) were gradually mixed in at 50°C using a mechanical stirrer. ZrB2 and SiC particles were also added to the resin solution, stirred for uniform distribution. Carbon fabrics, cut to 100*100 mm^2, were impregnated with this resin mixture. Post-impregnation, the samples were heated at 80°C for 30 minutes to eliminate the remaining solvent and prevent bubble formation. The pre-coated layers were stacked, molded, and cured at 90°C under 300 bar pressure for two hours, followed by a post-baking process at 140°C and 300bar for two hours. Three-point bending and tensile tests were conducted according to ASTM-D790 and D3039 standards. Thermal properties were evaluated with a maximum temperature of 3000°C by ASTM-E285-80 standard.

4. Results and Discussion

Sample G3 has the lowest insulation temperature, dropping below 150 degrees Celsius by 60 seconds (Figure 1). Lower weight carbon fabrics have higher thermal conductivity than heavier ones, enhancing heat transfer from the flame to the back of the insulation. Additionally, finer weave and lower density fabrics allow heat to pass more easily

According to Table 1, the greatest reductions in weight and thickness are seen in samples G1 and G3, respectively. When exposed to an oxyacetylene flame, sample G1's Novolac epoxy resin pyrolysis, forming char, which reacts with oxygen.

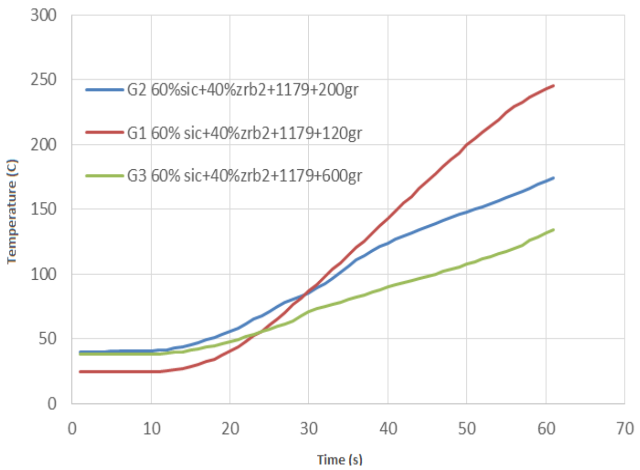

Figure1. Backside temperature of samples during the oxyacetylene test

Sample G1, with a 120g fabric weight, shows the least weight loss post-test. Lighter fabrics can absorb resin better and allow deeper penetration of ceramic particles than heavier fabrics. The increased void space in lighter fabrics facilitates better resin infiltration and saturation. Additionally, ZrB2/SiC particles penetrate more deeply among the fibers of lighter fabrics. The lighter fabrics reduced thickness results in more layers being destroyed by ablation, while weight and fabric structure significantly influence ablation depth.

Table 1. Oxyacetylene Test Result

SAMPLE	WEIGHT LOSS (%)	THICKNESS REDUCTION (MM)
G1	15.3	2.09
G2	21.3	1.02
G3	21.6	1.34

Figure-2a shows areas of ceramic particle agglomeration. In regions where the ceramic layer has eroded, some ceramic particles remain clustered on the carbon fabric, with residual ash visible, while the carbon fibers maintain good alignment.

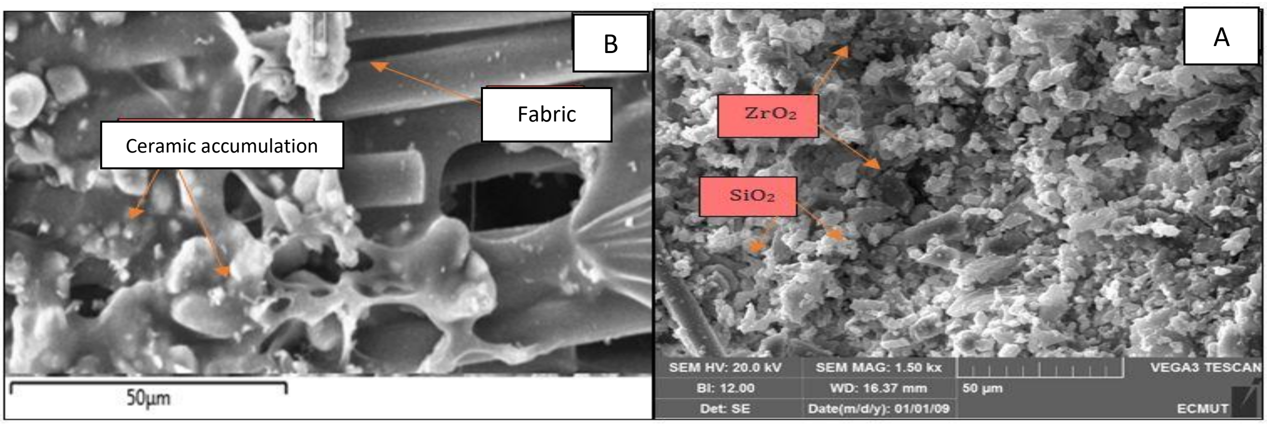

Figure2. SEM analysis after oxyacetylene test

Figure-2b indicates that the ceramic layer effectively reduces surface erosion and preserves the fabric's structure by preventing heat and oxygen penetration. However, areas where ceramic particles have eroded show severely damaged carbon fabric layers.

The tensile and bending test results (Table 2) shows that the elastic modulus and bending modulus increase with increasing fabric weight in the composite.

Table 2. Mechanical Evaluation Results

SAMPLE	TENSILE STRENGTH (MPA)	ELASTIC MODULUS (GPA)	BENDING STRENGTH (MPA)	BENDING MODULUS (GPA)
G1	321.3	8.78	410.25	87.46
G2	410.9	10.57	565.11	74.19
G3	468.35	14.75	677.18	89.43

5. Conclusions

Composites containing 60% SiC and 40% ZrB_2 ceramic particles were evaluated for mechanical properties and erosion resistance. The sample with 600g fabric exhibited the highest tensile and bending strength. The incorporation of SiC and ZrB_2 particles improved the erosion resistance of the insulation. SEM analysis revealed that in regions with exposed ceramic particles, the carbon fibers remained well-arranged, indicating the ceramic layer's effectiveness in reducing surface erosion and maintaining fabric cohesion by preventing heat and oxygen penetration. The oxyacetylene test showed that sample G1, with 100g fabric, had the least weight loss (12.94 grams) but the deepest pyrolysis zone (2.09 mm) due to increased flame penetration. Sample G3 had the lowest backside temperature, remaining below 150°C after 60 seconds. XRD analysis indicated that phases such as ZrO_2, B_2O_3, $ZrSiO_4$, and SiO_2 significantly impacted the composites' erosion resistance.

6. References

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