



High temperature ablation and thermo-physical properties improvement of carbon fiber reinforced composite using graphene oxide nanopowder



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ABSTRACT

Ablative nanocomposites were prepared by incorporating graphene oxide (GO) nanosheets into resole type phenolic resin and then impregnating them into rayon-based carbon fabric. GO were dispersed into phenolic resin at 0.25, 0.75, and 1.25 wt.% loadings using high shear mixing to insure uniform GO dispersion. The morphology and structure of graphene oxide nanosheets (synthesized by a Hummers method) and the structural, thermal and ablative properties of the samples were characterized using Fourier Transform Infrared spectroscopy (FTIR), X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), oxyacetylene flame test, and Thermo-Gravimetric Analysis (TGA) methods. It was found that the thermal stability and ablative properties of the GO/phenolic resin/carbon fiber composites were greatly enhanced due to the well dispersion of GO sheets in polymer matrix and the strong interfacial interaction between the GO sheets and phenolic matrix as well as layered carbon structure after pyrolysis. The specimen with 1.25 wt.% GO showed the best results in the rate of ablation, thermal diffusivity and thermal stability compared to other samples. This laminate had constant thermal diffusivity at different temperatures, and improved char yield and ablation rate to about 10% and 51%, respectively.

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1. Introduction

Charring ablation materials is the first choice for the thermal protection system of a vehicle subjected to severe aerodynamic environment during hypersonic reentry [1–3]. This protective function is accomplished by a self-regulating heat and mass transfer process known as ablation [4–6]. In the ablation process of a charring material, the high heat flux is dissipated by endothermic thermo-chemo-mechanical phenomenon. In these processes, gaseous products are released and porous char is left [7–9]. The char region plays an important role in thermal properties of ablative composite, because of surface protection performance of char layer as a secondary insulator [1,6].

Experimental evaluations have revealed that carbon reinforced phenol-formaldehyde resin (PR) which is considered as the most diffused charring ablative material is superior to the others [10]. The matrix with carbon fibers is less oxidized, and almost no change of shape is observed [11]. A lot of research has been developed in both thermal insulation and char structural integrity

carbon fiber/phenolic resin composite with the introduction of nanofillers [6,12–15]; For example carbon nanofibers (CNF) cause major changes in the heat transfer rates and affect the resultant combustion chemistry [16]; Nano silica loaded resin forms a thin and uniform layer on the fabric surface. Nano silica layer on the carbon-fabric resists the slippage of different layers against applied shear forces. Enhanced interlaminar shear strength makes the composites more resistant to the high aerodynamic shear forces and is expected to result in enhanced ablation resistance. Nano silica loading increases the number of grain boundaries, resulting in lower thermal conductivity. Reduced thermal conductivity also assists in increasing the ablation resistance as the heat front advancement will be slowed down there by allowing the phenolic resin to hold the fabric layers together for longer durations [17]. Multiwall carbon nanotube (CNT) nanocomposite displays enhanced flexural and shear properties. Well dispersed CNTs present in nanocomposite, are strengthening the fiber–matrix interface by bridging the interfacial micro cracks leading to enhanced interlaminar shear strength [18]. Phenyl Polyhedral Oligosilsesquioxane (Ph-POSS) with large phenyl groups can enhance the rigidity and carbon contents to form a thick carbon layer resulting in the high flame resistance and the small mass

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ablation [19]. Comprehensive study on montmorillonite (MMT) organoclay, CNT and POSS shows that lower loadings of CNT and POSS in polymer resin provide similar performance as that of higher loading of MMT organoclays [20]. CNTs also are easier to produce and therefore are available in much larger commercial quantity at a more affordable price than POSS. For this reason, recent publications have focused on the improvement of phenolic composites ablation resistance with the addition of CNTs [14,21].

In comparison with quasi-1-D CNT, graphene with 2-D lattice of carbon possesses similar mechanical performance but larger surface areas. Thus, graphene nanoplatelet is expected to be a good candidate for being used as a synergist agent in ablation composites. Despite the improvement in thermal properties, graphene nanoplatelets are difficult to disperse in phenolic matrix. In this work, we intend to produce graphene oxide (GO) due to the impressive performances of GO, including good dispersion and strong interactions between GO and phenolic matrix [22] and investigate effect of GO on the thermal and ablation properties of composite.

2. Experimental

2.1. Materials

A resole type phenolic resin (IL-800/2, Rezitan Co., Tehran, Iran) was selected as a polymeric matrix because of the high oxidation resistance and heat of ablation, having a high char yield (55–60%) at temperatures above 650 °C in inert atmosphere and a uncured low viscosity. Fig. 1 shows the differential scanning calorimetric analysis of the uncured resole type phenolic resin in nitrogen. This curve shows that the pre-cure and cure temperature of phenolic resin are about 120 and 160 °C, respectively [23].

Carbon fabric (T300, DY3K-200-T model made in Japan) was used as reinforcement. Dispersed graphene oxide (GO) in water used as thermal improvement filler was prepared from crystalline graphite (ROTH-1-7614, ROTH Co., Germany) using Hummers' method [24]. Properties of these materials from Refs. [25,26] and datasheet are given in Tables 1 and 2.

2.2. Sample preparation

In this study, weight (or volume fraction) of woven carbon fiber in the composite is kept constant to find out the effect of graphene oxide on the thermal and ablation properties. The woven carbon fiber/matrix weight ratio for the composites is around 65/35

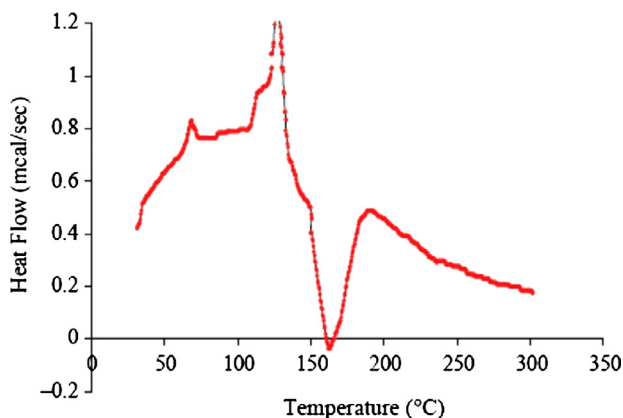


Fig. 1. Differential scanning calorimetric of the uncured resole type phenolic resin in nitrogen [24]. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

(43/57 v/v). The GO/resole/carbon fabric composite which contained 0, 0.25, 0.75 and 1.25 wt.% of GO with respect to matrix weight fraction was referred to as RG0, RG1, RG2 and RG3, respectively. To prepare sample, Phenolic resin was diluted using acetone by mechanical mixing. The necessary amount of phenolic resin and nanofiller particles were mixed and stirred for 30 min and 1000 rpm. Then 10 layers of carbon cloth impregnated by this modified phenolic resin as matrix by hand lay-up in steel mold by 100 × 100 × 2.5 mm dimensions. The curing process for the samples involved 3 temperature steps according to the DSC analysis (Fig. 1): 80 °C for 30 min, followed under hot press by 110 °C for 1 h and then 140 °C for 30 min. After curing, the composite was post-cured for 0.5 h at 150 °C. The composition and properties of the samples at room temperature condition are given in Table 3 to analyze the back temperature and surface erosion in oxyacetylene flame test and to measure thermal conductivity.

2.3. Characterization

Apparent density and porosity of the composite laminate was determined with submerging in water according to ASTM D-4018 (Table 3).

X-ray powder diffraction (XRD) patterns were obtained using a Miniflex X-ray diffractometer (the Netherlands) equipped with CuK α radiation, in the 2 θ Bragg–Brentano geometry.

Fourier transform infrared (FTIR) spectra were collected at a resolution of 4.0 cm⁻¹ on a Bomem SRG1100G S/N spectrometer at 25 °C in the range of 4000–400 cm⁻¹.

Ablation performance and thermal behavior of the samples were characterized by the oxyacetylene flame test (according to ASTM-E-285-80, external heat flux of 2.5 × 10⁶–10 × 10⁶ W m⁻² by hot gas 3400 K, Sample by 50 × 50 × 2.5 mm dimensions), thermogravimetric analysis (TGA, heating rate of 10 °C min⁻¹ under a nitrogen atmosphere) and measurement of the thermal diffusivity according to Bahramian et al. work [7]. For measuring thermal diffusivity, the sample by 50 × 50 × 2.5 mm dimensions was placed into alumina foam cavity. Heat source was provided at the bottom of the cavity. The top of the cavity was insulated by the alumina foam to minimize heat transfer through the sample (to ensure one-dimensional heat transfer condition). Three K-type thermocouples, with an error of ±0.1 °C, were selected. One was attached to the back surface of the sample (cold surface), and the two others were recording the temperature of vessel and jacketing heater. All the thermocouples were connected to a PC-based data acquisition unit. The back surface temperature was recorded as it varied with time and data were obtained in the test duration time of 5 min. These data were used to calculate the thermal diffusivity according to inverse solution technique of heat transfer modeling.

The sample charring was investigated by Burning test. A thermogravimetric analyzer can also measure char yield but its disadvantage is that it can only work with small samples (0.5 mg), while burning test use 1 g and therefore provide for a more representative sample. The sample was placed in a ceramic dish inside a furnace where it was heated to 800 °C with heating rate of 30 °C min⁻¹ under a nitrogen atmosphere and then it remained in that condition for an hour. The char yield was determined using the difference in sample mass before and after test.

The morphology of the sample surface after oxyacetylene test was observed by scanning electron microscopy (SEM, JEOLJSM-5600LV).

3. Results and discussion

Fig. 2 compares the X-ray diffraction (XRD) patterns of graphite powder, graphene oxide, cured resole resin and nanocomposites

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