



The fabrication and performance of C/C composites impregnated with TaC filler



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ARTICLE INFO

Article history:

Received 11 August 2016
 Received in revised form
 28 November 2016
 Accepted 5 December 2016
 Available online 26 December 2016

Keywords:

C/C composite
 Fabrication
 Ablation
 Oxidation
 Filler

ABSTRACT

Carbon-carbon (C/C) composites are one of the most promising lightweight materials for hypersonic, rocket, aero and gas turbine engine applications. Yet, despite decades of research and development by the aerospace industry, C/C materials are still difficult and expensive to fabricate due to long processing times and other factors. In the present study, a low cost liquid resin re-infiltration and pyrolysis technique was used to fabricate complex shaped C/C components containing anti-ablation tantalum carbide (TaC) filler. Thermalgravimetric analysis (TGA) was used to investigate the material carbonisation process to determine the pyrolysis schedule. Scanning Electron Microscopy (SEM) and Raman spectroscopy was used to characterise the C/C material. The C/C material flexural strength, oxidation and ablation rates were investigated with three-point bend testing, exposure to a high temperature air oven and an oxyacetylene flame torch respectively. A flexural strength of 62 MPa was found after four re-impregnation and pyrolysis treatments. TaC fillers, impregnated in the C/C during densification, reduced the material ablative effects at ultra-high temperatures. The linear ablation rate for C/C impregnated with TaC was found to be superior to the C/C composite material without TaC additives.

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1. Introduction

C/C composite materials consist of high strength carbon fibre tows embedded in carbon matrix making them light weight, high-strength and high-temperature resistant materials [1,2]. Of particular significance is that C/C composites can withstand temperatures up to 2800 °C in many environments, above which their strength reduces due to thermal damage to the fibres. Subsequently, C/C composites are extremely important for thermal protection systems and are currently the most promising lightweight materials for hypersonic leading edges and engine components [3]. However, fabricating these materials is time consuming and costly. Other disadvantages include poor inter-laminar properties, the difficulty of making joints and the high oxidation rates in air at temperatures greater than 320 °C.

The C/C oxidation rates are controlled by the transport of gaseous species to and from the reaction front where both chemical reactions and gas-phase diffusion occur. Overcoming the high oxidation rates in C/C is challenging and is the focus of much study [4–8]. High oxidation rates occur typically along the fiber axis at

the fiber/matrix interface. Therefore, C/C composite materials exposed to high temperatures and oxidising atmospheres need to be protected.

Surface coatings are often used to protect C/C against severe oxidation and ablation during oxyacetylene flame testing [9–11]. However, cracking and spallation can occur in these coatings during thermal cycling due to thermal expansion differences between the C/C substrate and the coating. When the coating layers become damaged, oxidation protection is compromised and additional protection is needed to delay complete oxidative failure of the C/C composite. One method of this protection is to embed anti-oxidative fillers of various compounds in the carbon matrix during the fabrication process [12–14]. In air at high temperatures, these fillers function as oxygen getters that form protective glassy layers on the C/C surface to delay the ingress of oxygen to the substrate. In particular, boride filler is known [15,16] to form a glass phase that fills interspaces, cracks and pores in the C/C composites up to 1000 °C. However, above 1700 °C, many filler additives gasify before forming a stable glass to rapidly seal any breach of the C/C protective coating [2]. Current studies [17–20] suggest that the oxides formed from TaC coatings inhibit the oxidation in C/C under an oxyacetylene flame. In particular, the TaC to Ta₂O₅ transformation acts as a barrier against further erosion during

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oxyacetylene flame testing. Subsequently, the glassy Ta₂O₅, with melting point of around 1870 °C, is potentially a good candidate for the partial sealing of incipient cracks and pores that develop in C/C materials during high temperature ablation. To date, few publications on the effectiveness of TaC fillers in C/C composites are found in the literature.

In this study, C/C composites containing tantalum carbide filler were fabricated using liquid resin re-infiltration and pyrolysis techniques. Thermalgravimetric analysis was used to determine the optimal processing parameters during carbonisation. The TaC fillers were embedded in the C/C matrix and used as anti-ablatives during high temperature testing. The mass ablation, linear ablation and oxidation rates from C/C (as-prepared) and C/C-TaC (TaC-impregnated) materials were obtained. In addition, scanning electron microscopy, Raman spectroscopy, surface profilometry and flexural strength testing was carried out.

2. Fabrication

A PAN based carbon fibre reinforced polymer (CFRP) material was prepared using phenolic MTM080-S¹ pre-preg 2D-plane till weave fibre sheet with a 6 k fibre tow count. A hand lay-up, with stacking sequence ($\pm 45^\circ$, $0^\circ/90^\circ$) consisting of 20 plies, was used to form panel thicknesses of around 7 mm. The panels were cured in an autoclave overnight at a constant pressure of 300 kPa according to Table 1. Carbonisation of the CFRP material was achieved through pyrolysis processing for several days at high temperature using a tube furnace operating in a nitrogen gas environment at atmospheric pressure. Additional material fabrication details are published elsewhere [21].

The C/C material densification involved mixing SC-1008² liquid phenolic with and without TaC filler powders (10 wt % and particle sizes $\leq 6 \mu\text{m}$). Re-infiltration of the C/C composites involved submerging the samples in SC-1008 resin mixtures and placing them in a vacuum chamber where they were cycled several times from atmosphere to a pressure of -80 kPa . This removed any residual or trapped gases left behind in the composite pores. The TaC filler was impregnated during the final, less penetrative, re-infiltration stage. This allowed TaC particles to collect at the sample surface, away from the centre or bulk of the C/C material. The samples were then vacuum bagged and autoclave cured overnight at 620 kPa according to Table 2. Four re-impregnations and pyrolysis treatments at 900 °C were used to prepare, over a four week period, the fully densified C/C and C/C-TaC materials.

3. Experiment

3.1. Flexural strength testing

A load force was applied to several specimens through a three-point system at a constant rate of 0.5 mm/min. The three-point bend specimen lengths and widths were 30.0 mm and 10.0 mm respectively with thicknesses varying from 6.9 mm to 7.1 mm. The testing apparatus had a span of 28 mm, a loading nose of 6 mm and the support rollers were 3 mm in diameter in accordance with [22]. The three-point bend testing was performed at room temperature in air on several specimens until failure.

3.2. Material characterisation

Material analysis was performed using a Joel JSM-840 SEM and

Table 1
Curing parameters for MTM080-S phenolic resin.

Curing stage	Ramp rate (°C/min)	Temperature (°C)	Dwell time (hours)
1	2.0	95	0.5
2	2.0	160	1
3	2.0	120	2
4	2.0	60	NA

Table 2
Curing parameters for SC-1008 phenolic resin.

Curing stage	Ramp rate (°C/min)	Temperature (°C)	Dwell time (hours)
1	1.0	50	2
2	0.5	70	2
3	0.5	120	2
4	0.5	0	NA

intermetallic compounds present on the surface were identified using Energy Dispersive Spectroscopy (EDS). An optical microscope was used to examine the sample surfaces after flame and hot air oven testing. Analysis of area fraction porosity and TaC particle distribution was conducted using ImageJ software (version 1.44p) where optical images from several sample cross-sections were used to obtain the average area fraction values.

Raman spectroscopy was conducted using a Thermo Scientific DXR Dispersive Raman confocal microscope with a 532 nm laser. The analysis was performed by examining small particles from the composites using a 50 times magnification objective and a 50 μm slit aperture. The laser power was set to 5 mW and 30 scans were averaged with an exposure time of 1 s for each scan. The low resolution grating was employed providing a spectrum with a resolution of 2 cm^{-1} .

An NETZSCH TG 209 F1 thermalgravimetric analysis (TGA) system was used to investigate CFRP sample weight losses for the SC-1008 and MTM080-S phenolic resin systems. The analyser consisted of a high-precision balance with a pan that was loaded with a few milligrams of the sample. The sample was placed in a small electrical oven where it was heated from room temperature to 800 °C at a rate of 2 °C/min and monitored via a thermocouple. The atmosphere in the oven was purged with nitrogen gas to prevent the CFRP samples from oxidising and it should be noted that the fibre mass of the sample was assumed to be non-reactive during the pyrolysis.

A MarSurf XC 10 profilometer interfaced with evaluation software was used to examine the front sample surfaces after flame testing. The drive unit consisted of a tracing arm in which a stylus, with a measuring force of 40 N, was traced over a distance of 55 mm. The stylus had an accuracy of 1 μm in measuring direction and the profile measurements were performed across the central crater regions of the flame tested samples.

3.3. High temperature testing

Oxidation testing was performed on several samples that were placed in a 1200 °C pre-heated air oven for up to 4 min, removed and then left to cool naturally in air.

Oxyacetylene flame testing was conducted on several samples using the setup shown in Fig. 1. Sample areas and thicknesses were approximately 65 × 65 mm and 6.9–7.1 mm respectively. The testing involved positioning the samples 500 mm away from the flame nozzle and moving them slowly toward it before reaching a fixed distance of 105 mm from the nozzle and stopping for 60 s. A 2 MW/m² heat flux was used. The heat flux was calibrated using a Vatell Corporation circular foil heat flux transducer (Model No.

¹ UMECO fibre product: ACG, T300 2/2 Twill Carbon Fibre, MTM080-S Phenolic.

² DURITE binding agent: SC-1008 phenolic resin.

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