

Fabrication and erosion studies of C–SiC composite Jet Vanes in solid rocket motor exhaust

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Abstract

C–SiC composite Jet-Vanes were fabricated using liquid silicon infiltration and tested in a plume of aluminum based solid propellant to study erosion resistance. The Jet-Vanes showed excellent resistance to thermo-oxidative erosion; average linear and mass erosion rates were 1 mm/s and 5 g/s, respectively. Morphology of the eroded surfaces suggests that alumina particles in the plume hit the leading edge of the Jet-Vane, damage it and some material is eroded away. Residual silicon melts and reacts with oxygen to form silica which in turn reacts with SiC matrix. The matrix of SiC, silicon and un-reacted carbon is loosened and erode by high shear forces. Once carbon fibers get exposed directly to the plume, these may be both eroded and oxidized.

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1. Introduction

C–SiC composites are considered as potential materials for advanced aero-engines, Jet-Vanes, leading edges and nose-tips of re-usable space vehicles.¹ These composites are lighter than the refractory metals like tungsten and hence provide higher payload capacity of the aerospace vehicles. The Jet-Vane material must have good erosion resistance to the particulate flow in addition to high thermal shock resistance i.e. high thermal conductivity, low CTE and good strength. Erosion of Jet-Vanes depends on the fiber architecture, fiber to matrix ratio and testing conditions. Though numerical simulation studies have been reported to assess thermal response,² there is no mathematical model to predict erosion for a given composite, experimental method is the only way to study the erosion rate and its mechanism.

Liquid silicon infiltration based 3D stitched C–SiC composites have excellent thermal and mechanical properties^{3–5} and thus would have good erosion resistance to solid rocket motor (SRM) plume due to oxidation resistant hard SiC matrix and third direction stitching.

Aim and scope: In this study, typical sized C–SiC composite Jet-Vanes were fabricated and exposed to SRM plume to investigate erosion resistance. The erosion morphology and mechanisms are discussed.

2. Experimental

2.1. Fabrication of Jet-Vanes

Fibrous preforms of near net shape Jet-Vanes were prepared by stitching several carbon-fabric layers of the 3k 8H-satin with 6k carbon fiber tows. Number of stitches was maintained in the range 350–400 per 100 cm². The preforms were rigidized by vacuum infiltration of coal–tar pitch at 200–300 °C followed by carbonization at 900–1000 °C, and graphitization at 2400–2600 °C in the nitrogen atmosphere. The rigidized

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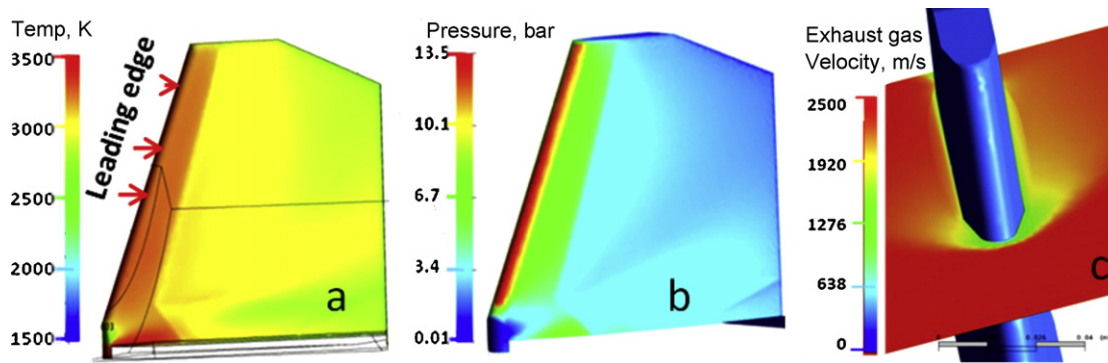


Fig. 1. Plume temperature, pressure and velocity profile over surface of Jet-Vane.

preforms were further densified by a hot-isostatic-pressure-impregnation-carbonization (HIPIC) process at 700–800 °C and 1000 bar. These were termed as C–C preform blocks; their density was found to lie in the range 1.55–1.60 g/cm³. This fabrication procedure has been reported elsewhere also.^{3–5} The C–C preform blocks were machined to typical sized Jet-Vanes and were siliconized under vacuum at 1450–1650 °C for about 10,000 s, density 2.2–2.3 g/cm³. The siliconized products (C–SiC composites) were grinded with diamond wheels to get the final dimensions of the Jet-Vanes. Chemical composition of the final product was also determined using acid digestion method.⁶

2.2. SRM testing

Erosion test of the Jet-Vanes was carried out under a hot-firing test using SRM. The SRM was loaded with the composite propellant composed of hydroxyl-terminated poly-butadiene, aluminum powder (17%, w/w) and ammonium per-chlorate (oxidizer) and some other additives. Using a special fixture four Jet-Vanes were held at 90° w.r.t. each other in the SRM plume. The plume hits the leading edge of the Jet-Vane at 0° for 8 s; the plume is parallel to the fabric layers and perpendicular to the third direction (reinforcement) stitches.

Conditions in the vicinity of the Jet-Vanes were determined using commercial Fluent software. CFX-5. It solves 3-D Reynolds Averaged Navier Stokes equations on unstructured grid with $k-\epsilon$ turbulence model. The wall boundary condition was implemented through Log-law, the inlet boundary condition is fixed at the rocket motor chamber condition and a supersonic outflow boundary condition is used at the outlet of the nozzle. At the inlet of the nozzle pressure and temperature were assumed uniform. A log-normalized residue of $1e-04$ was taken as the convergence criteria. Estimated plume temperature at the stagnation point of the leading edge of a Jet-Vane is about 3500 K; stagnation pressure is about 13 bar and velocity parallel to the surface of the Jet-Vanes is about 2000 m/s (Fig. 1). Velocity and temperature gradients in the direction parallel to the surface are relatively negligible as compared to the corresponding gradients in the perpendicular direction to the surface.

The equilibrium composition of the plume gases was calculated using a code from NASA “Chemical Equilibrium with

Applications”.^{7,8} The code determines chemical equilibrium product concentration, based on the free-energy-minimization principle. Theoretical composition of the gas is estimated in terms of mol fractions as H₂ = 0.347; CO = 0.252; HCl = 0.148; Al₂O₃ = 0.088; N₂ = 0.076; H₂O = 0.073; CO₂ = 0.009; H = 0.004, Cl = 0.001; OH = 0.0002; O₂ = 0.00002. The density of the plume gases is about 2.57 kg/m³. Uniformity of gas composition and flow properties may deviate little bit during the test.⁹

2.3. Characterization

Dimensions and weight of the Jet-Vanes were measured before and after the test using micro-calipers and microbalance. The surface morphology and microstructure were also observed using FEI Quanta 400 Scanning Electron Microscope.

3. Results and discussion

3.1. Properties of Jet-Vanes

It was found that the Jet-Vanes have un-reacted carbon (12–15%), residual silicon (8–12%) and SiC matrix (28–30%) by weight. Physical, mechanical and thermal properties of the 3D stitched C–SiC composites were also determined and have been reported already^{3–5}; these are also summarized here: fiber content = 40–42% (v/v); flexural strength = 160–240 MPa; tensile strength = 70–100 MPa; Young’s modulus = 40–45 GPa; strain at failure = 0.15–2.0%; Vickers hardness = 2800 g/mm²; fracture toughness = 5–6 MPa/m^{1/2}; in plane and through thickness thermal diffusivity = 75–15 and 35–8 mm²/s, respectively (temperature range 25–1500 °C); in plane and through thickness CTE = 1×10^{-6} and 2×10^{-6} /°C, respectively (temperature range 25–1050 °C).

3.2. SRM testing

During the test, surface of the Jet-Vanes attains the plume temperature quickly and it might lead to spalling of the layers due to mismatch in the expansion of different constituents of the Jet-Vanes. However, all the Jet-Vanes remained intact and the fabric layers of the Jet-Vanes did not separate after

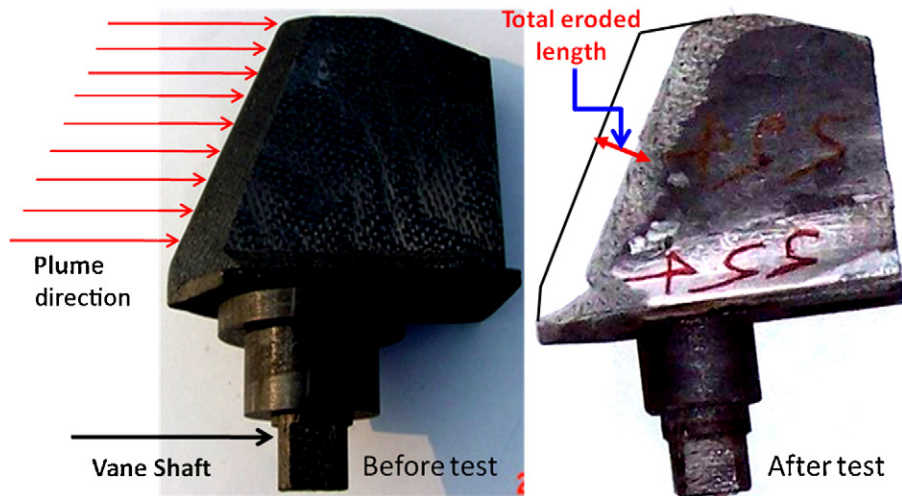


Fig. 2. Typical Jet-Vane photograph before and after SRM test.

the test because of adequate through-thickness reinforcement (Fig. 2). The overall mass erosion rate was about 5 g/s. The erosion length (the linear eroded distance perpendicular to the leading edge of the Jet-Vane; total eroded length is shown in Fig. 2) varied with position, and the erosion pattern was similar in all the Jet-Vanes. Erosion length was determined using coordinate measuring machine by comparing initial and after-test geometries of the Jet-Vane. Total erosion length was maximum (about 12 mm) at the tip of the leading edge and least near the shaft, and it is about 8 mm. Unequal erosion w.r.t. location of the Jet-Vane is due to the variation in the thickness of the Jet-Vane.

3.3. Microstructure

3.3.1. As fabricated Jet-Vanes

In addition to the carbon fibers, three phases viz. SiC matrix, residual silicon and un-reacted carbon matrix can be observed (Fig. 3). SiC is uniformly distributed in the matrix

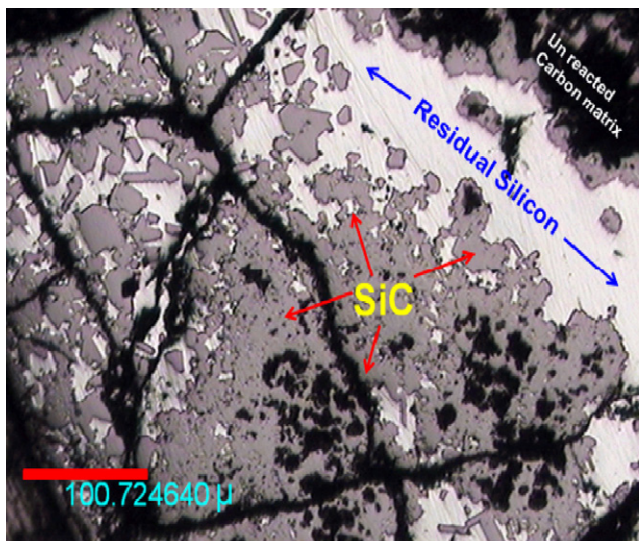


Fig. 3. Microstructure of C-SiC composite Jet-Vane before test.

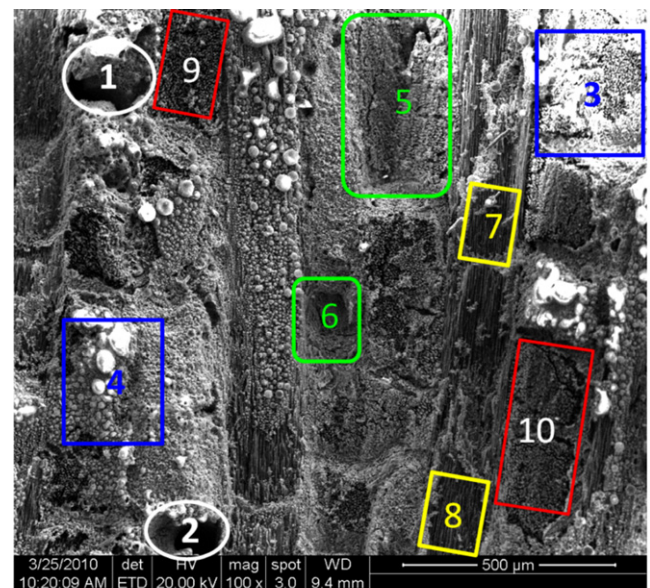
and some particles of SiC are also observed isolated in the un-reacted residual silicon pool. Similar microstructures have been reported in the literature.^{10,11}

3.3.2. Eroded leading edge surface

The extent of erosion of different constituents is not uniform (Fig. 4). The fibers show more erosion as compared to the matrix; also the un-reacted carbon matrix eroded much more than the SiC matrix. Whitish spherical particles of different sizes were also observed on the eroded surface.

3.3.3. Side (flat) surface

Overall thickness of the Vane was observed to be somewhat higher than that before the test. A thick whitish layer on both



Location 1 & 2- Silicon evaporation; 3 & 4 – Deposited alumina
5&6- Matrix chunk removal; 7&8- Eroded warp fibers;
and location 9&10- Eroded weft fibers

Fig. 4. Jet-Vane leading edge microstructure after SRM test at low magnification.

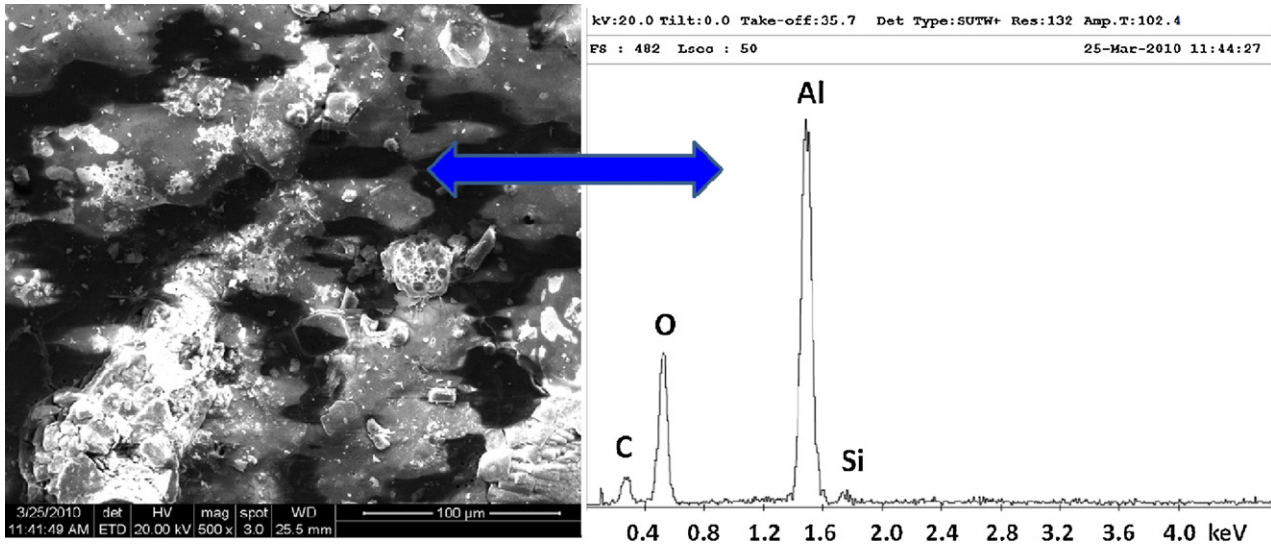


Fig. 5. Microstructure and EDAX of Jet-Vane flat surface after SRM test.

the side surfaces was observed (Fig. 5). The EDAX and XRD revealed that the layer is made up of alumina (Figs. 5 and 6). It is also observed that some particles of the carbon fibers and SiC crystals are embedded into the layer. Microstructure of the leading edge after the test was further studied in detail.

3.4. Erosion mechanism

In the C–SiC composites, the carbon fibers are well covered with the matrix composed of SiC, un-reacted carbon matrix and residual silicon. The plume hits the leading edge of the Jet-Vane in the direction parallel to the plane of the fabrics and perpendicular to the third direction stitches holding the fabric stack. The plume consists of a very large mass flow rate of very hot gases and alumina particles.

One or more of the following phenomena may be taking place simultaneously.

- (a) The Jet-Vanes get heated due to the surrounding plume and also face the impact of alumina particles. The leading edge is greatly damaged and some material also flies off.

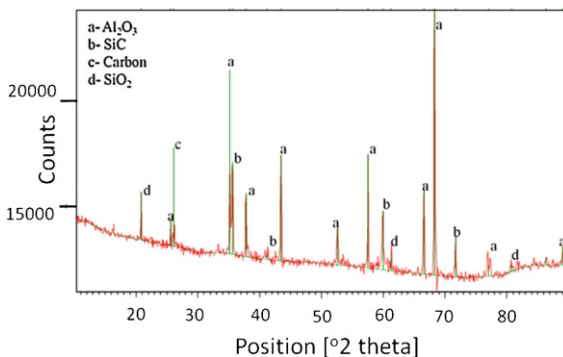


Fig. 6. XRD of eroded surface.

- (b) The composite – fabrics and the matrix – tends to expand, mainly in the direction of the thickness of the fabric stack.⁵ Although there is a large mismatch in the coefficient of thermal expansion (CTE) of the composite (Fig. 7) in-plane and through-thickness directions, the fabrics are held together by the third direction stitches.
- (c) The residual silicon in the matrix melts and diffuses towards the outer surface. A fraction of the silicon evaporates, while another fraction reacts with oxygen in the plume and forms SiO₂ above 1000 °C.¹² Some voids get created in the matrix.
- (d) The carbon (in the matrix and the fibers) is thus exposed and reacts with oxygen in the plume.
- (e) The silica formed reacts with SiC matrix and forms gaseous products above 1430 °C¹³; further, a gap between the fabric layers and the matrix gets created which loosens and the matrix gets removed.
- (f) Once some matrix gets removed, the carbon fibers are exposed leading to their oxidation; fiber diameter decreases and the composite weakens locally and it leads to further erosion.

The overall damage of the Jet-Vanes may be attributed to the cumulative effect of the following factors, in order of their importance.

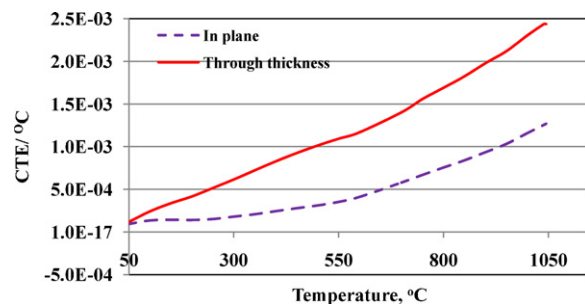


Fig. 7. CTE vs. temperature of 3D stitched C–SiC composite.

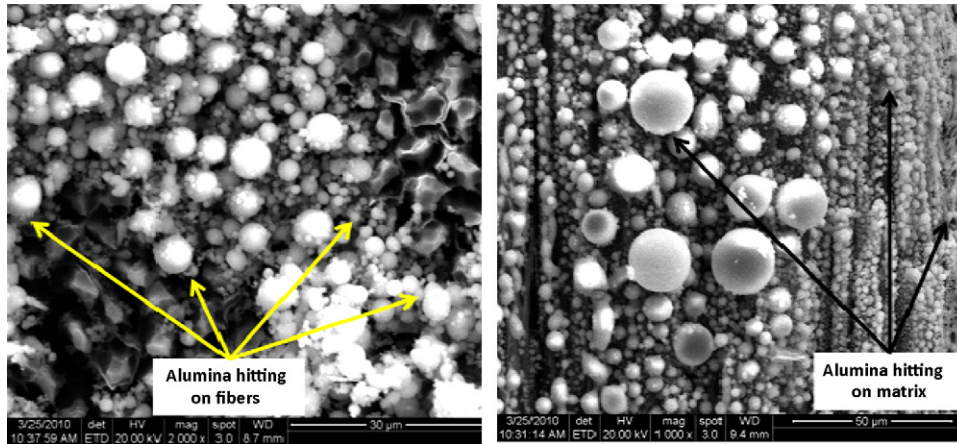


Fig. 8. Erosion of C–SiC composite constituents by alumina particles hitting.

- (i) Impact of alumina particles,
- (ii) chemical erosion and
- (iii) loss of loosely bound material because of large mass flow rate of gases.

3.4.1. Particle erosion

The hot alumina particles impinge on the hot material surface causing mechanical damage to the fibers and to the matrix (Fig. 8). The damage is more on the leading edge. The plume contains Al_2O_3 in solid as well as in liquid state.^{7,8} Alumina particles in the plume may be of different sizes (1–100 μm).^{7,8,14} Mass of a particle is proportional to cube of its diameter. Bigger particles cause greater damage. However, smaller particles close to the surface deflect and go around the Vane and are deposited along with liquid alumina on the flat surface of the Vanes. On the other hand, the bigger particles move straight depending on their inertia and are hardly affected by the change of the flow field. Due to particle momentum, material removal takes place. It is observed that the

removal of the carbon fibers is higher as compared to the SiC matrix. Fibers get damaged and their strength decreases. SiC matrix is hard and is not easily damaged.

3.4.2. Chemical erosion

It is reported that OH, H_2O , CO_2 , O_2 are the primary aggressive species in the SRM plume for chemically attacking carbon fibers and un-reacted carbon matrix of the composite.^{14–17} The following chemical reactions are envisaged between the plume components and the constituents of the C–SiC composites:

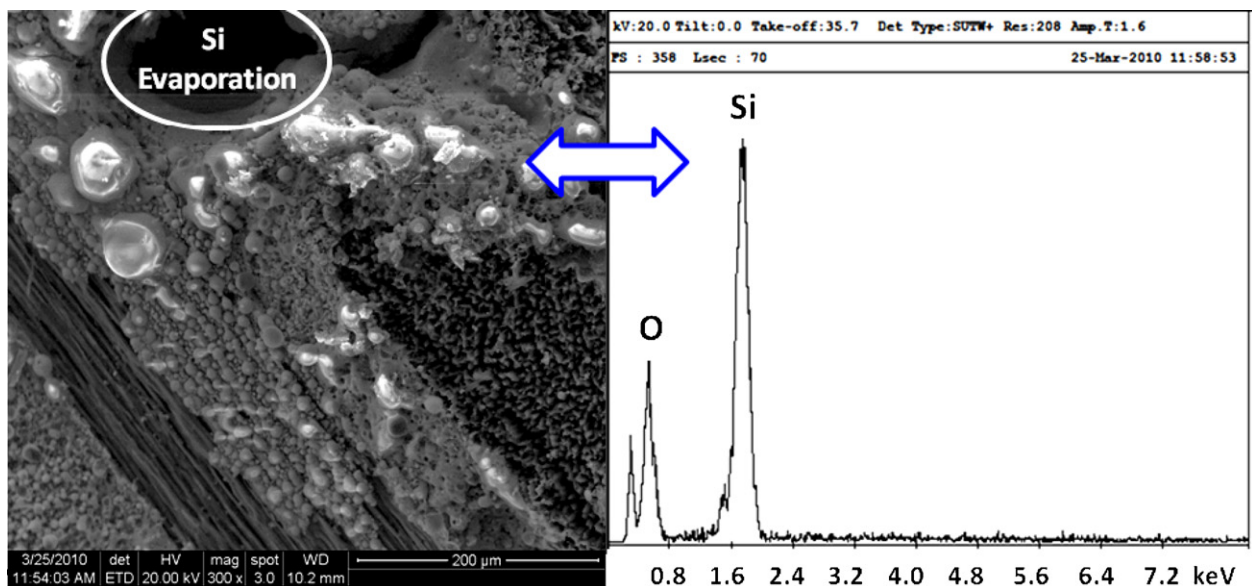


Fig. 9. Erosion due to residual silicon and oxidation of SiC matrix.

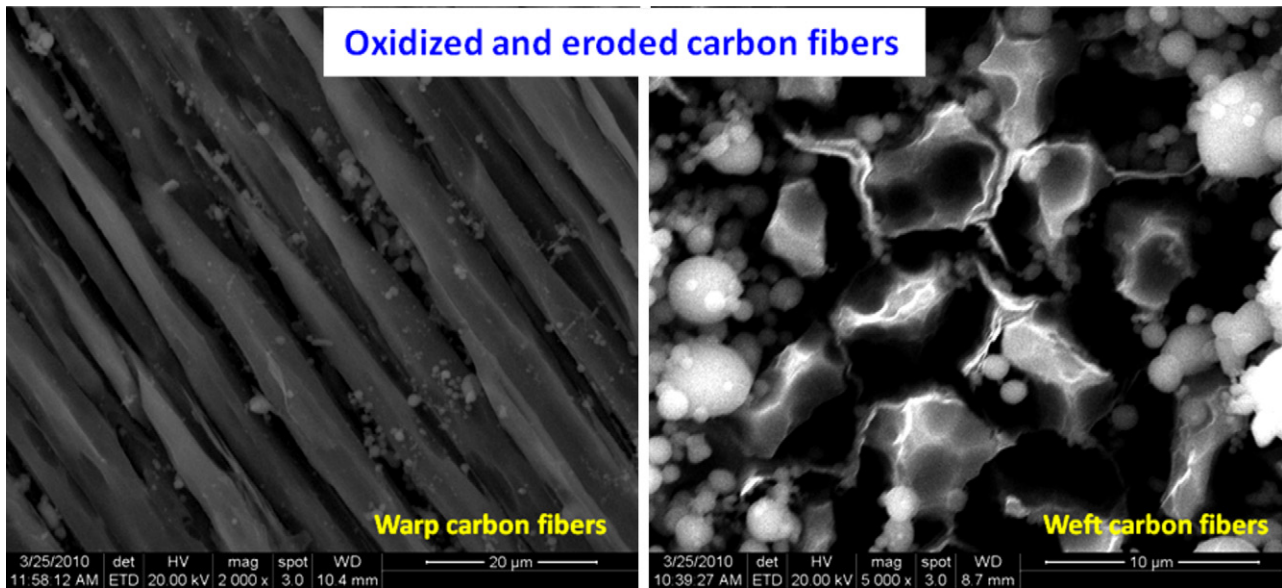
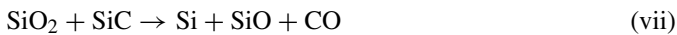


Fig. 10. Oxidation and erosion of carbon fibers.



Carbon has a poor oxidation resistance even at temperatures as low as 700 K^{14–16}; thus it may be safely assumed that all the reactions (i)–(iv) are taking place at the plume temperature. The carbon fibers are well covered by SiC matrix, therefore ablation of carbon fibers and SiC matrix in C–SiC composites is different from mere oxidation. For oxidation of carbon fibers removal of SiC is essential which may be either by chemical erosion or by flow.

For chemical erosion of SiC, reactions (vi) and (vii) need to take place. SiC reacts with oxygen at temperature above melting point of silicon and forms amorphous silica¹⁸ which in turn reacts with another molecule of SiC and forms SiO and CO. Silica also forms due to presence of residual silicon. Above 1410 °C, residual silicon melts, diffuses toward the surface of the Vane, evaporates and is oxidized to SiO₂ which deposits on the surface. EDAX analysis shows mainly Si and oxygen, suggesting that there should be SiO₂ (Figs. 6 and 9).

In the absence of SiC and residual silicon, the carbon fibers react with H₂O vapor and oxygen. In addition to chemical erosion, the fibers encounter impingement by alumina particles and this leads to their enhanced erosion. Thus the fibers at the leading edge are damaged and tapered because of the combined action of chemical erosion and due to flow leading to mechanical erosion (Fig. 10). Similar findings have been reported in the literature.^{19,20} When there is only oxidation, the tail end of the carbon fibers becomes flat-bottomed¹⁹; when ablating, those are tapered.²⁰ Thus thermo-physical and thermo-mechanical attacks cause ablation.²¹

3.4.3. Mechanical erosion: loss of loosely bound material

In the plume, the flow is complex and unsteady. In Jet-Vanes erosion due to flow erosion is also observed (Fig. 11).

The binding strength at the interface (between the matrix and the reinforcing fabrics) has a large influence on the resistance to erosion. Once the matrix gets loosened due to melting or softening of residual silicon and removal of un-reacted carbon matrix, an additional portion of the matrix gets removed purely due to shear force. However contribution of such erosion is relatively small as the composite has reinforcement in all the directions but relatively low in through thickness direction. The vortex could increase erosion because of enhanced shear stress in the local region. Fig. 11 shows the cavity due to excess erosion caused by vortex at the leading edge tip. It is also observed that the fibers have eroded much more than the matrix.

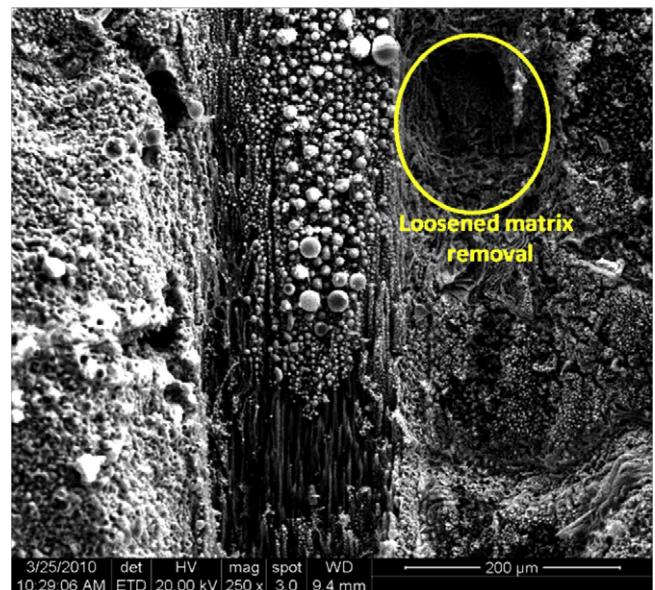


Fig. 11. Mechanical erosion of loosened matrix due to vortex formation.

4. Conclusions

- 3D stitched C–SiC composite Jet-Vane shows excellent thermal shock and erosion resistance under SRM.
- Stitching prevents fabric layers to get separated even under large shear force and very high CTE in the through thickness of the composite.
- Erosion pattern is similar in all the Jet-Vanes; however it varies from 1 to 2 mm/s depending upon the location.
- Morphology of the eroded surfaces suggests that alumina particles in the plume hit the leading edge of the Jet-Vane, damage it and some material is eroded away. Residual silicon melts and reacts with oxygen to form silica which in turn reacts with SiC matrix. The matrix of SiC, silicon and un-reacted carbon is loosened and erode by high shear forces. Once carbon fibers get exposed directly to the plume, these may be both eroded and oxidized.
- Erosion of Jet-Vanes may be further lowered by reducing residual silicon and un-reacted carbon fraction in the composite and by improving fiber reinforcement network in the through-thickness direction.

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