# **Ceramic coating on flexible external insulation blankets for reusable missions**

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Flexible thermal protection material (flexible external insulation; FEI) is used in the Reusable Launch Vehicle-Technology Demonstrator (RLV-TD) leeward region where multiple curvatures are present. FEI configuration has silica cloth layers on either side with Cerablanket felt sandwiched and stitched together using quartz thread. FEI should ensure an airframe skin temperature in the leeward region within the temperature constraint limits (100°C) of aluminum alloy during the RLV-TD ascent (4.8 W/cm<sup>2</sup>) and descent (3.7 W/cm<sup>2</sup>) phases. A ceramic room-temperature coating with low surface solar absorptivity ( $\alpha$ ) and high thermal emissivity (*ɛ*) to generate the required thermal gradient at the backwall was developed. The salient features of the ceramic coating are roomtemperature curability, ease of application, minimum weight, durability, erosion resistance, stable optical characteristics, easily repairable, non-contaminating, high purity and high temperature compatibility with silica fabric. The properties of the ceramic coating are weight/area: 0.025-0.030 g/cm<sup>2</sup>, solar absorptivity the UV-VIS region: 0.12-0.18, IR emittance: >0.86, backwall temperature: <120°C (from kinetic heating simulation test).

**Keywords:** Emissivity, flexible external insulation, protective ceramic coating, reusable missions.

#### Introduction

THERMAL protection systems are essential for the successful launch and operation of all spacecrafts – manned and unmanned. During re-entry of the vehicle into the Earth's atmosphere at a velocity exceeding 17,000 mph, the aero-thermodynamic interactions with the atmosphere can produce extremely high surface temperature, which is well above the melting point of metals. Hence, special thermal materials/shields are required to protect the vehicle and its components/payloads. Although the vehicles are built using highly advanced construction methods and materials, the airframe is formed primarily from aluminum alloy and it can only withstand up to 350°F without softening. The selection of a thermal protection system

depends upon the mission trajectory of the vehicle. In order to meet the mission objectives and design criteria, the thermal protection system must be composed of appropriate materials whose selection is based on heat dissipation needs and environmental constraints. As a whole since the design cannot be optimized from the point of view of each component, a compromise must be reached based on the requirements. Mission environment consists of three major regimes: the launch environment, the space environment, and atmospheric re-entry. In the launch environment, the vehicle is subjected to liftoff and ascent acceleration loads, vibration, aerodynamic loads and aerodynamic heating, shock, acoustic loads and loads imposed by flow of liquid fuel and sloshing. In addition, vehicles may encounter natural conditions such as wind, rain, hail, lightning, and salt water<sup>1</sup>. The natural environment of space includes vacuum, ionizing radiation, spacecraft charging, contamination, degradation due to UV radiation, existence of atomic oxygen in the upper atmospheric layers, and impact from meteoric debris. The re-entry environment imposes the most severe aerodynamic heating in addition to shock and acoustic loads. Similar to the launch environment, the re-entry environment also includes natural conditions such as wind, rain, hail, sand and dust<sup>2,3</sup>. Heat produced by different means is dissipated by several methods -(i) Heat sinks where a high thermal conductivity material absorbs the heat and distributes it quickly and uniformly away from the part of the spacecraft it was designed to protect<sup>4,5</sup>. (ii) Active cooling - In high heat flux areas, fluids can be used as a liquid or gaseous heat sink when distributed to hot sections via a cooling loop. The proposed cooling fluids include liquid metals like sodium or potassium as well as gaseous hydrogen cooling<sup>6</sup>. (iii) Transpiration cooling – which involves the ejection of a fluid or gas through a porous skin into a boundary layer between the heat flux and the surface, thus reducing the adiabatic wall temperature of the surface<sup>1,2,7</sup>. (iv) Radiation cooling – here most of the heat flux is reflected back towards the blackbody of space by a high emissivity coating on the protected substrate. (v) Ablation-it is an effective mechanism of minimizing the total energy that the vehicle absorbs. Thus, the heat is expended in a material phase change rather than being conducted to the interior of the vehicle.

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Advanced flexible reusable surface insulation (AFRSI) blankets were installed on the orbital manoeuvring system (OMS) pods of orbiter vehicle Challenger. These blankets consisted of quartz fiber insulation batting encased in woven quartz fabric and quilt sewn with Teflon-coated quartz thread. Post-flight analysis of STS-6 showed that extensive damage occurred on ten blankets with significant loss of the cover fabric on 20 other blankets. Similarly post-flight analysis of STS-7 revealed that 20 blankets on the right OMS pod suffered damage, which included missing fabric and eroded batting. It was noted from the analysis that flight damage was caused due to particulate impact and aerodynamic effects. There was no evidence of significant thermal exposure above 650°C during the two missions. Over 371.6 m<sup>2</sup> of AFRSI blankets was installed in the orbiters Atlantis and Discovery covering different portions of the vehicle. Based on laboratory wind tunnel data, there was considerable concern on the durability of insulation. Thus a programme was initiated to develop and evaluate a protective coating for AFRSI<sup>8,9</sup>

The leeward region of the Reusable Launch Vehicle-Technology Demonstrator (RLV-TD) has multiple curvatures and hence flexible thermal protection systems (TPS) capable of withstanding high temperatures are required. Flexible external insulation (FEI) is required for RLV-TD to restrict the airframe temperature to less than 100°C. In order to achieve the temperature constraint, it is proposed to bond FEI blanket on the leeward surface of RLV-TD. The leeward region of RLV-TD experiences aeroheating during both the ascent and descent phases. Various options in the design of FEI were studied by the project design team (Aerothermal Simulation and Testing Division (ASTD)) of Vikram Sarabhai Space Centre (VSSC), Thiruvananthapuram as it was a challenge to attain the temperature constraint by preventing the thermal energy thus absorbed from reaching the backwall for a duration of 880 s. For RLV-TD HEX mission, it was decided to accept the configuration of FEI, which has silica cloth layers on either side with Cerablanket felt (density 128 kg/m<sup>3</sup>) sandwiched and stitched together using quartz thread (thickness of FEI is 15 mm). So the TPS system design is required to generate the desired temperature gradient at the backwall by a suitable configuration of FEI having a ceramic surface layer coating with high emissivity and low absorptivity. To meet such requirements, a high-purity ceramic coating having high temperature compatibility to silica/quartz fabric, improved adhesion, flexibility, stable thermo-optical properties, minimum weight, brushable/sprayable grade and which is room temperature curable and repairable is required. The present study was aimed at developing a ceramic room-temperature coating with low surface solar absorptivity and high thermal emissivity to generate the required thermal gradient at the backwall. This article details the processing parameters of the coating and its characterization. The FEI blanket was

# Experiment

### Materials

Silica binder (M/s ALDRICH, Mumbai) and silica powder (M/s Surabhi Industries, Pune) were used for the coating. FEI blanket was supplied by Advanced Materials and Ceramics Division (AMCD), VSSC which was waterproofed/bonded by Polymers and Special Chemicals Division (PSCD) before applying the ceramic coating.

The optimization of the binder-to-solute ratio needed several iterations, so as to get a low absorptivity, high emissivity ceramic coating which is compatible with that of the silica substrate. Silica is known for its low thermal conductivity and high emissivity. However, the selection of a suitable binder to fulfill the requirements like minimum weight, being room temperature curable, stable optical properties, etc. was a major challenge. Based on the literature review of high emissivity coatings for TPS materials, efforts were made in-house to select a suitable silica binder so as to assure the required properties. A large number of samples of different dimensions  $(35 \times 35 \text{ mm})$ ,  $25 \times 25$  mm,  $150 \times 150$  mm,  $300 \times 300$  mm) and shapes (square, rectangular, curved) were coated with the ceramic slurry to optimize on various factors such as feed ratio of binder to filler, absorptivity, emissivity, coating weight, curing time, number of passes of coating, etc.

The finalized stoichiometric quantity of silica binder and silica powder was weighed into a polythene jar and approximately 20 wt% of ceramic balls was added to it. The mix was milled in a ball mill for 1-2 h. The product was subsequently stored in a closed container. As a process check prior to application of the coating, viscosity of the slurry was measured using Ford cup (B-4) and found to be in the range 15-25 cps. The slurry was then charged into a spray gun and sprayed over the hardware. Along with the hardware,  $150 \times 150 \times 15$  mm and  $25 \times 25 \times 15$ 15 mm samples were also coated for absorptivity and emissivity measurements. The number of passes was decided according to the required weight gain to be achieved. The formulated coating was applied over the waterproofed/bonded FEI by spraying. The coating was left in open air for 48 h for complete curing, taking care to avoid dust deposition before final weighing.

#### **Results and discussion**

Figure 1 shows the optical images of the silica binder taken inside the quartz tube, glass and silicon wafer at various stages of the curing process. The binder consisted of uniform dispersion of micron-sized spherical beads of silica particles in water.



On glass substrate; magnification: 50X

On silicon wafer; magnification: 50X





Figure 2. (a) Raman and (b) X-ray diffraction of flexible external insulation solution after RT curing.

Figure 2 *a* and *b* shows the Raman and X-ray diffraction (XRD) patterns of the ceramic coating solution taken after room temperature curing for 48 h, which confirms the presence of Si–O–Si linkages. The novelty of the coating is in the selection of its ingredients in such a way as to obtain a high emissivity coating when cured, which is compatible with that of the silica substrate. The viscosity of the slurry measured using Ford cup (B-4) is found to be in the range 15–20 cps. The coated sample exhibited solar absorptivity in the range 0.12–0.18 in the UV-VIS region and IR emittance >0.86. Weight/area of the ceramic coating was 0.025-0.030 g/cm<sup>2</sup>.

### Kinetic heat simulation test

Kinetic heat simulation (KHS) test was carried out to evaluate the thermal response of the blanket system to the estimated heat flux history from trajectory. The peak heat flux experienced during the ascent phase was 4.8 W/cm<sup>2</sup> and during re-entry it was 3.7 W/cm<sup>2</sup> (Figure 3). The test



Figure 3. Heat flux history at the leeward surface.



Figure 4. Backwall temperature of five different samples.

was successfully completed with a maximum backwall temperature of 73°C against the allowable 120°C (Figure 4). The coating was intact and no cracking or peeling-off was observed after the test. Figure 5 shows the FEI blanket during KHS test.

#### Acoustic test

This test evaluates the integrity of FEI TPS under direct acoustic impingement (157 dB). FEI blankets on curved panel were tested at National Aerospace Laboratories



Figure 5. Ceramic coated FEI during kinetic heat simulation test.



Figure 6. FEI curved panel undergoing acoustic test at National Aerospace Laboratories, Bengaluru.

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(NAL), Bengaluru and then using non destructive technique (NDT) method in VSSC (Figures 6 and 7). Posttest NDT results for bond integrity and FEI quality were found to be acceptable.

#### Vibration test

This test was carried out to evaluate the integrity of FEI TPS under vibration loads. The test conditions were: random: 13.5 g (in all axes), sine: longitudinal: 20 mm



Figure 7. Non-destructive technique inspection for bond integrity after acoustic test.



Figure 8. *a*, FEI vibration test article on shaker; *b*, panel undergoing vibration test.

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DA (10–16Hz), 10 g (16–100 Hz); lateral: 12 mm DA (10–16 Hz), 6 g (16–100 Hz) and sweep rate: 2 Oct/min (Octave/minute) (Figure 8 a and b). The integrity of FEI after vibration test was found to be normal.



Figure 9. Testing radio frequency performance during KHS test.



Figure 10. Backwall temperature during the radio frequency performance test.



Figure 11. Ceramic-coated FEI after thermo-vac test.



Figure 12. Depressurization rate (a) and thermo-vacuum cycling (b).



Figure 13. Specimens before and after shear flow test.

### RF transparency test

This test was carried out to evaluate the radio frequency (RF) performance and voltage standing wave ratio (VSWR) of telemetry antenna under FEI with heating conditions. Maximum aero-heat flux was simulated on

snout of leeward region. Peak heat fluxes – ascent:  $4.8 \text{ W/cm}^2$  and during re-entry:  $3.7 \text{ W/cm}^2$  were applied. Figure 9 shows the testing of RF performance during the KHS test and the Figure 10 shows backwall temperature graph. Thus the ceramic coated FEI could ensure the temperature constraint limit of  $100^{\circ}$ C in the leeward

region for the RLV-TD and the functional performance of telemetry with ceramic coated FEI under the flight thermal environments is qualified (VSWR  $\leq 2$ ).

#### Thermo-vac test

This test evaluates the integrity of FEI TPS under thermovacuum conditions. The minimum pressure requirement for thermo-vacuum cycling is  $10^{-5}$  mbar followed by depressurization at maximum rate and three temperature cycles from 0°C to 100°C with 1 h soak time and 1 h rising time. FEI blanket of size 300 mm × 300 mm bonded to aluminum sheet after ceramic coating (Figure 11) was subjected to evacuation (up to  $6 \times 10^{-1}$  mbar) for 30 min using rough pumping system, and was subsequently brought back to ambient conditions. The specimen was tested successfully. Figure 12 shows the plots of depressurization and thermo-vacuum cycling. Post-test NDT results for bond integrity and FEI quality were found acceptable.

## Shear flow test

This test evaluates the integrity of FEI TPS under shear loads. The test was carried out over six FEI specimens of size  $42 \text{ mm} \times 35 \text{ mm}$ . Maximum shear stress of 113 Pa was simulated at Mach 3 in supersonic wind tunnel of heat transfer facility. No peeling-off was observed for all the six specimens at the shear stress test conditions (Figure 13).

#### Conclusion

A room-temperature curable silica-based ceramic coating has been developed which is compatible with silica or/and quartz fabric. The coating can be applied over FEI blankets either by spraying or brushing with minimum weight gain.

The coating could withstand a maximum heat flux of  $4.8 \text{ W/cm}^2$  during the KHS test, vacuum of  $10^{-5}$  mbar fol-

lowed by depressurization and three temperature cycles from 0°C to 100°C with 1 h soak in thermo-vac test and maximum shear stress of 113 Pa without any peeling off in the shear flow test. Thus the coating successfully passed the vibration and acoustic test and qualified for RLV-TD.

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ACKNOWLEDGEMENTS. We thank the authorities of VSSC, Thiruvananthapuram for permission to publish this work. We also thank our colleagues at the Analytical and Spectroscopy Division, VSSC for the thermal, chemical and spectral analysis and other testing agencies for the functional tests of the samples.

doi: 10.18520/cs/v114/i01/137-143