Experimental analysis of formulation and coating process of LDAM for thermal protection systems

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ABSTRACT

KEYWORDS

Thermal Protection Systems,
Low Density Ablative Material,
High Velocity Low Pressure,
Dry Film Thickness (DFT),
Back Wall Temperature (BWT),
Taguchi,
ANOVA.

The Thermal Protection System (TPS) is a membrane used to avoid aerodynamic heating of metallic and plastic surfaces during flight of missile. Between a heat source and the protected entity, the thermal protection system is interposed. Although the Orbiter's re-entry surface heating is primarily convective, ample energy in the air molecules disassociated from the shock layer and given the potential for additional heating. Various composite materials are used in thermal insulation systems to achieve higher backwall temperatures in order to maintain high temperatures and pressure upon re-entry. A Low-Density Ablative Material (LDAM) is prepared and coated using the High Velocity Low Pressure (HVLP) method of coating. The process of applying the chemical composition of the present invention shall ideally require spraying at room temperature, without the need to add heat to the treatment of the composition in order to affect the repair and to obtain the optimum dry film thickness specifications. In this present research, the configuration of the Taguchi experiments is considered and a series of experiments are carried out for the coating process using the L9 orthogonal array by varying the control factors such as air pressure, spray time and nozzle size (lock no.) and the performance parameters are optimized such as Dry Film Thickness (DFT) and Back Wall Temperature (BWT) using Taguchi optimization technique to get the optimal input levels. Finally, a validation test is carried out according to the optimal levels obtained from the optimization technique of Taguchi and the most influential parameters are found using ANOVA.

1. Introduction

The Thermal Protection System (TPS) is the barrier used to protect the metallic and composite surfaces from aerodynamic heating during flight of missile and to protect from the heat and cold of space while in the orbit. Fig. 1 shows vehicle re-entry from space. Re-entry of the orbit heating differs from the normal atmospheric heating associated with jet aircraft and this is governed by TPS design and their characteristics. Fig. 1 shows the Vehicle / orbiter re-entry from the space.

The orbiter returned to the atmosphere as a blunt body by having a very high (40^0) angle of attack, with its broad lower surface facing the direction of flight. In compliance with the simple thermodynamic relationship between pressure and temperature, about 80% of the warm orbiter at re-entry is caused by compression of the air infront of the hypersonic vehicle. A hot shock wave is produced infront of the spacecraft, which deflected much of the heat and prevented the surface of the orbiter from peak heat directly.

Fig. 1. Vehicle re-entry from the space.

Re-entry heating is largely convective heat transfer by superheated plasma between the skin of the orbiter and shock wave. For this a very low-density material is used to protect the orbiter against this type of heating.

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The design of thermal protection systems is intended to have a smooth, aerodynamic surface while shielding the metal structure from high temperatures. The charges incurred by the device include launching acoustics, aerodynamic heating, related structural deflations and changes in orbital temperature as well as natural environmental conditions such as salt fog / mist, wind and rain. In addition, when the Orbiter detached from the external Tank, the TPS had to tolerate pyrotechnic shock loads. The TPS consists of a range of materials added externally to the exterior structural skin of the Orbiter to passively preserve the skin at appropriate temperatures, especially during the re-entry process of the mission.

1.1 Coatings

An overlay is a coating which is generally referred to as the substrate on the surface of a material. The persistence of smearing the coating can be decorative, practical or both. The layer itself may be an all-over coating, covering all the substrate or it may cover only portions of the substrate. Functional coating may be added for the purpose of changing the surface properties, such as adhesion, weather tolerance, corrosions or wear resistance on substrate. In other situations, such as the manufacturing of semiconductor devices in which the substratum is a wafer, the coating introduces absolutely a new property, such as a magnet reaction or electrical conductivity.

There are several types of TPS materials are used on the orbiter to protect the external surface. The materials include tiles, advanced flexible reusable surface insulations, reinforced carbon-carbon and flexible reusable surface insulation and ablative material coatings. All such surfaces have been coated with high emissivity to guarantee the optimum rejection of convective heat by the vinyl additive. Each type of TPS had specific heat protection, impact resistance, and weight characteristics, which determines the locations where it is used and how much quantity is used.

In this research, ablative material is considered for coatings of the orbiter and it is made of a resinous composite material that slowly vaporizes the heat during descent and allowing the heat to dissipate along with the ashes. The ablative coating materials have the vinyl, silicon, hardeners and solvents with the thermal insulation properties. These heat shields are used in all early NASA missions. Fig. 2 shows ablative composite materials.

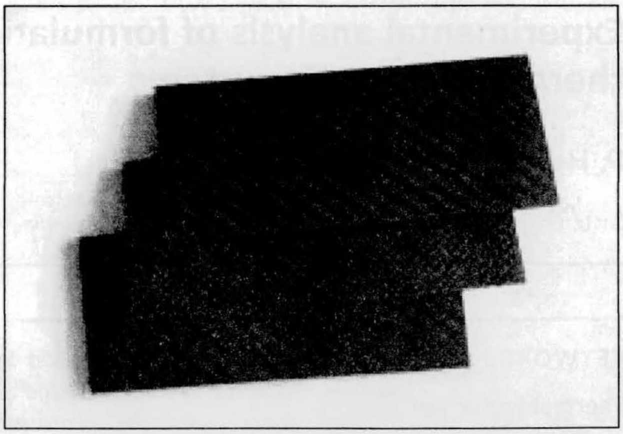

Fig. 2. Ablative composite material.

2. Literature Review

Beth (1992) have discussed some of the VOC prevention methods used in surface coating facilities include powder, waterborne radiation-curable and high-solids coatings and also reviewed many coating facilities able to reduce VOC emissions by converting conventional spray, airless, or air-assisted airless equipment to electrostatic or High-Volume-Low-Pressure (HVLP) units. The proposed modifications and product substitutions can reduce the labor expenditures, decrease consumption of coating materials and reduce air pollution has defined. Jeffs (2000) have proposed few adjustments in HVLP spray guns for effective use of Auto Refinishing Shop to ensure maximum transfer efficiency and high-quality results. For that, he is decreased the distance amongst the spray gun and the targeted location and slightly increased the speed of the stroke. He always ensures that the best transfer efficiency by keeping the spray gun square and at a consistent distance from the target surface throughout the stroke.

Liang (2016) have studied the ablative insulation materials such as glass fiber / phenolic resin, carbon fiber/phenolic resin to isolate heat flux into internal structures of rocket motors to remove much heat by mass consumption materials. Barnwal and Bissa (2015) have reviewed the different factors which affect the thermal coating process. In his studies, he observed that the need of TBCs like thermal barrier coatings improves the life of implements and increases performance of hot section. The improvements in the properties of coatings are mainly due to mismatch at interface, decrease in thermal conductivity and porosity, which lower the absorption of corrosive chemicals at the interface.

Rallini et al. (2019) studied the various non polymeric ablative materials and the versatility of their thermal properties by doing thermal analysis of the ablatives such as differential scanning calorimetry, thermo-gravimetric analysis, thermo mechanical analysis. Jamaluddin (2015) have studied the characteristics of the conventional paint spray compared to the High-Volume-Low-Pressure (HVLP) type of paint sprays used in the automotive painting industries in terms of cost and air quality in the spray booths. Wijewardane and Gowswamy (2012) have studied the emission of thermal radiation from a surface can be controlled spectrally and spatially, and the polarization and coherence of the radiation field for possible new energy-related applications. He also suggested the common design and fabrication methods for future research opportunities to optimize the paints and coatings for spectral selectivity applications.

From the literature review, the following objectives are considered for the present research work:'

- To study and choose the best thermal protection system in terms of material, cost and its mechanical properties for aerospace applications.
- To formulate the Low-Density Ablative Material (LDAM) and applying the LDAM using High Velocity Low Pressure (HVLP) spray coating process on the substrates.
- To optimize the process parameters on performance characteristics and mechanical properties of the obtained material using Taguchi technique.
- To find out the most influential process parameters using ANOVA

3. Methodology

Taguchi's strategy is an effective method for the plan of a brilliant framework. It gives, a productive as well as an orderly way to deal with improve plans for execution and quality. Moreover, Taguchi parameter configuration can lessen the change of frame work execution. Selection of an appropriate orthogonal array for the experiments done on the basis of number of process parameters and its levels. As number of parameters 3 and number of levels 3, L9 orthogonal array is selected based on Taguchi design of experiments. Table 1 shows the process parameters and their levels.

Table 1

Process parameters and their levels.

Parameters	Level 1	Level 2	Level 3
Air pressure (Bar)	2	3	4
Spray time (Sec.)	1.2	1.4	1.6
Nozzle size (Lock No.)	20	26	32

Note: Based on the lock no. size chart the size of the nozzle is selected.

3.1 Signal-to-Noise ratios (S/N ratios)

The transformation of response values to S/N ratios is the initial step. For the computation of S/N ratios, equations of 'Larger the better', 'Smaller the better' and 'Nominal the better' are used. Subsequent analysis is carried out on the basis of these S/N ratio values.

a) Larger the Better:

$$\frac{S}{N} = -10\, log(\frac{1}{R}\sum_{j=1}^{R}\frac{1}{y2_{ij}}) \quad ...(1)$$

b) Smaller the Better:

$$\frac{S}{N} = -10\, log(\frac{1}{R}\sum_{j=1}^{R} y2_{ij}) \quad ...(2)$$

c) Nominal the Best:

$$\frac{S}{N} = -10\, log(\frac{1}{R}\sum_{j=1}^{R}(y_j - y_i)^2) \quad ...(3)$$

Where,

Y_{ij} is the value of the response

'j' in the i^{th} experiment condition, with i=1, 2, 3, ...n; j= 1,2...k

3.2 Analysis of variance (ANOVA)

It is a set of mathematical models used in analysis of the differences between group means and their related estimating procedures (such as the "variation" amongst and between group variations).

The statistician Ronald Fisher developed ANOVA. The ANOVA is based on the law of total variance, where the measured variance in a given component is separated into elements due to various variation sources. ANOVA offers a predictive measure in the simplest form as to whether two or more population means are the same and thus generalizes t-tests over two forms.

4. Low-Density Ablative Material (LDAM)

LDAM is made by using vinyl terminated silicone resin reinforced with hollow glass microspheres. The raw materials which are used for the formulation of LDAM are shown in Table 2.

- **Vinyl Silicon Resin:** Vinyl is a strong, durable, abrasion and moisture resistant with stand rust and corrosion; is electrically non-conductive and has excellent fire performance properties. Vinyl material is thermostatic materials and cure solely by solvent evaporation. Silicone is a widely used material in the aerospace industry due to its sealing properties, stability across an extreme temperature range, durability, sound dampening and anti-vibration qualities, and naturally flame-retardant properties.
- **Hardener:** Hardener is a member of many combination types. In certain blend hardener, the strength of the blend is simply improved, and is used as curing portion for other mixtures. In the chemical reactions during mixing phase a hardener can be either a reactor or a catalyst. Hardeners should often be used for their intended uses to make epoxy resin beneficial. Epoxy hardeners are typical examples of an hydride, amines, polyamide, aliphatic and cycloaliphatic.
- **Hollow Glass Microspheres (HGM):** HGM sometimes referred as micro balloons or glass bubbles and their diameters ranging from 10 to 300 micrometres. Hollow spheres are used in composites like syntactic foam and lightweight concrete as lightweight filler. Micro-balloons provide syntactic foam light weight, low thermal conductivity and a compressive stress resistance that is far superior to other foams.
- **Solvents:** Solvents are mainly two types they are aliphatic solvents and aromatic solvents. Gasoline and kerosene are examples of aliphatic hydrocarbon solvents. Common aliphatic hydrocarbon solvents used in paints and coatings are mineral spirits, hexanes and heptane's. An aromatic solvent is an aromatic hydrocarbon solvent containing naphtha, toluene or xylene. Aromatic solvents are mainly used in diverse manufacturing fields as solvents and diluents.

Table 2

Raw Materials used in LDAM formulation.

S. No.	Raw material	Weight
1	Vinyl Silicone Resin (Mixed with catalyst)	100±1gm
2	Hardener	10±0.1gm
3	Hollow Glass Microspheres (HGM)	40±1gm
4	Solvent (Hydrocarbon based)	150 to 175 ml

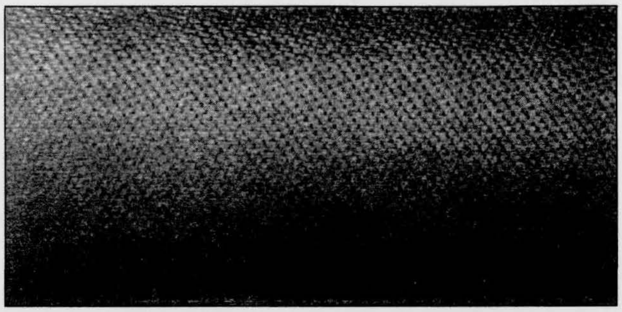

Fig. 3. Carbon phenolic material.

4.1 Workpiece material

Carbon phenolic is used as workpiece material in the present work and is shown in Fig.3. Carbon-phenolic composites are meant for heat protection of the aerospace structures like aircraft skins, nozzles and heat shields during the aerodynamics loading conditions. Carbon phenolic composites are made of combining two materials one is low density, rigid, carbon fibres impregnated in the phenolic resin.

4.2 Surface preparation before coating

Around 80 percent of paint failures occur either from inadequate surface preparation or from poor application of paint. No matter how good the color is it won't work to the full degree on or whether it's poorly prepared. Prior to surface preparation, visual inspection should be done on all steel products. This specifies the appropriate surface preparation techniques and the level of pre-preparation criteria for the relevant structure and the procedure should take place before the main preparation of the soil.

Pre-cutting can require grinding or filling processes to eliminate laminations, flammable edges and sprinklers of solder (ISO 8501-3) and/or to round or smooth corners and solder. The technique avoids all apparent weak points or defects to boost the surface to ensure a smooth film construction. Various surface preparation levels can be accomplished by diffent methods of cleaning. For this coating process following procedure should followed for the preparation of the coating.

1. Surface is visually inspected for defects like cracks, grooves and undulations etc.
2. If defects are found to be less than 01mm, it can be filled during spray process otherwise it has to be informed and take corrective measures.
3. The surface to be coated is rubbed by emery papers of 80 & 200 to remove, foreign materials and to create roughness.

Surface should be cleaned by acetone solvent. The cleaning should be done by using cotton cloth wetted with acetone. The surface is wiped till no grease or dust is found. The cleaning of substrate is checked by Whitman filter paper (grade 2). If no oil or dust are found on the filter paper then the surface is considered to be clean and should be completely dried for 30-45 minutes at room temperature.

4.3 HVLP coating process

Fig. 4 reveals the HVLP spray gun, which is similar to a standard spray gun using an air supply pump, but the spray gun itself allows a Lower Pressure Gun (LP) and a higher volume (HV) of air is required for aerosolising and propelling the paint at a lower atmospheric pressure. With decreased overspray, content consumption, and air emissions, the effect is a greater proportion of paint hitting the target surface. For the HVLP spray pistol, a regulator is often needed to reduce the air pressure from a traditional compressor. Alternatively, a turbine unit usually featuring a motor originating from a vacuum cleaner) may be used to accelerate the air without the need for an aircraft to operate on the compressor.

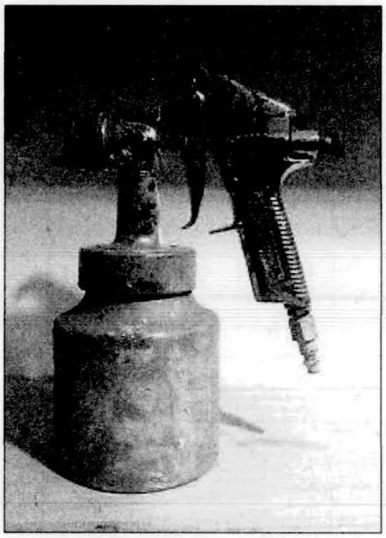

Fig. 4. HVLP spray gun used to spray LDAM.

4.4 Preparation of LDAM

1. The shelf life of all the raw materials shall be checked prior to mixing.
2. Resin and HGM mentioned in Table 2 is to be taken in a SS bowl (1 litre) add 100 ml of solvent and mix with spatula for 8-10 minutes.
3. Add hardener in the above mixture and mix thoroughly for 5-7 mins.
4. The viscosity of the mixture should be 20-30 sec by B3 cup viscometer.
5. LDAM is sprayed on the object by using HVLP spray gun. The air pressure should be maintained at 4-5 bar.

The following precautions to be taken care during spraying process:

1. LDAM is applied in four coats longitudinally from left to right to the motor to build up thickness up to 1.0 to 1.5 mm. For each coat minimum 15 to 20 minutes gap shall be given.
2. Similarly, the thickness can be increased by layer by layer as per the requirement.
3. Each layer thickness can be 0.5 to 1.0 mm. ensure complete curing of each layer as per acceptance criteria.
4. Specimen shall be prepared with LDAM coating under identical conditions to study the curing and adhesion.
5. If any roughness is observed, the surface can be rubbed with emery paper to level it.

5. Experimentation

5.1 Dry Film Thickness (DFT)

Fig. 5(a) indicates the outline of the spray region which is an ellipse and can also be distorted under the non-perpendicular structure of the spray gun to the target surface as seen in Fig. 5(b). Moreover, with spray angles and the stand-off gap between the pistols and the target surface, the spray coverage often improves. The spray region (A) can therefore be expressed geometrically:

$$A = \frac{\pi * Z^2 \tan \alpha \tan\beta}{\cos \theta} \quad ...(4)$$

where Z, α, β and θ are the standoff-distance of the spray gun, the span angle to the main axis, the span angle to the minor axis and the angle of inclination of the spray gun, respectively as shown in the Fig. 5 (a) and (b).

During the spraying process certain assumptions are made that there is no drag and that the atomization distribution over the spray is consistent. It can be implied that the coating layer thickness is uniform. Based on the assumptions, the spray area expressed in equation 4 and the Dry Film Thickness (TDFT) can be parametrically calculated by:

$$T_{DFT} = K * \frac{m * \cos\theta}{\rho_{cm} * \pi * Z^2 * \tan\alpha \tan\beta} \quad ...(5)$$

Where; m, ρ_{cm} and k are weight of coating material in liquid solution, coating material density, and coefficient of spray coating process respectively.

At Z = 0,2 m, α = 25,38°, the direction and angle were constant. β= 13.34 degrees and θ= 45 degrees. These values are based on process limits and the criteria for the spray gun.

5.2 Back Wall Temperature (BWT)

Back wall temperature is a temperature that will be assumed as an inner atmosphere temperature of re-entry vehicle.

At pyrolysis of composite will act at any point of time so that surface temperature does not exceed 400^{0}C at outer surface. But electronic packages inside the wall with stand upto 90^{0}C only. Fig. 6 shows the thickness of the substrate and coating LDAM. So, the temperature of the substrate is taken as a 773^{0}K and the coated surface temperature is calculated by

$$Q = \frac{A(T_2 - T_1)}{\frac{L_1}{K_1} \; \frac{L_2}{K_2}} \quad ...(6)$$

Where A is area, L_1 is thickness of the substrate, L_2 is the thickness of the coated LDAM on substrate, K_1& K_2 are the thermal conductivities (0.35 w/mm & 0.1 w/mm), T_1 & T_2 are the free surface temperatures and Q is rate of flow (10-20 w/mm²), T_1 is the 773^{0}k. These values are selected based on the process limitations and the specifications.

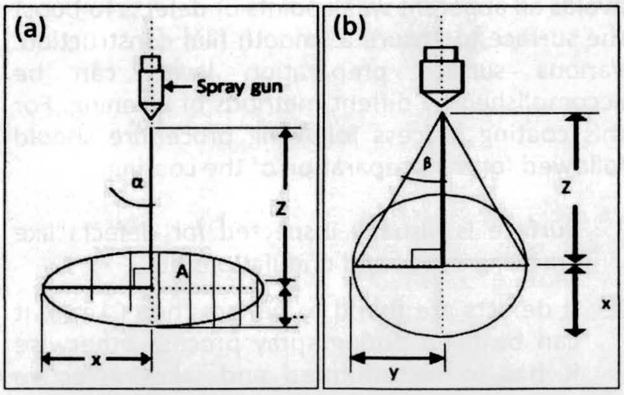

Fig. 5 (a) & (b). Span angles and standoff-distance in the spraying process (Front view and Side view).

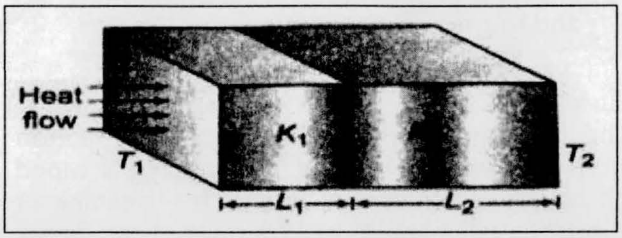

Fig. 6. Thickness of the substrate and coating LDAM.

Table 3

Responses of coated workpiece.

S. No.	Air Pressure (Bar)	SprayTime (Sec)	Nozzle Size (Lock No.)	DFT (mm)	BWT (°C)
1	2	1.2	20	0.571	157.2
2	2	1.4	26	0.646	149.7
3	2	1.6	32	0.793	135.0
4	3	1.2	26	0.933	121.0
5	3	1.4	32	1.011	113.2
6	3	1.6	20	0.841	130.2
7	4	1.2	32	1.379	76.4
8	4	1.4	20	1.131	101.2
9	4	1.6	26	1.256	88.7

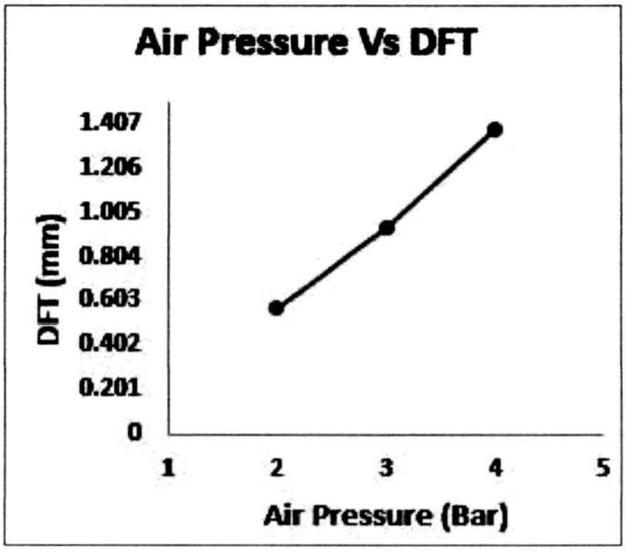

Fig. 7. Air Pressure Vs DFT.

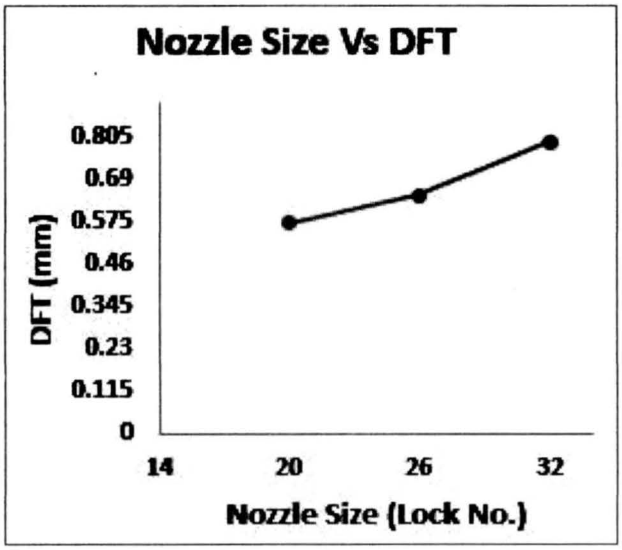

Fig. 9. Nozzle size Vs DFT.

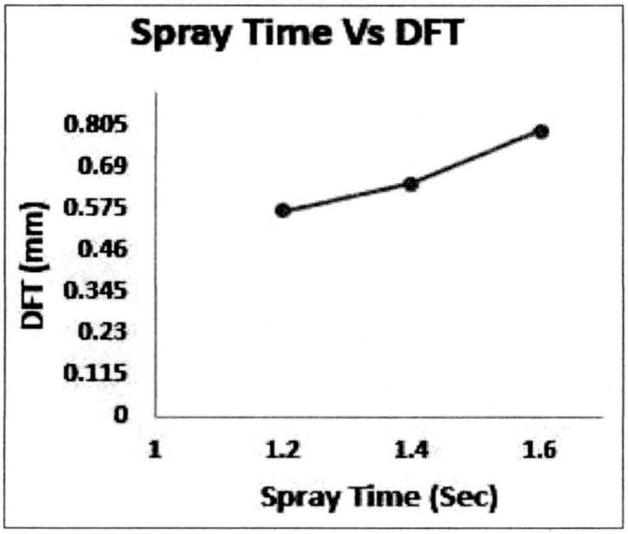

Fig. 8. Spray Time Vs DFT.

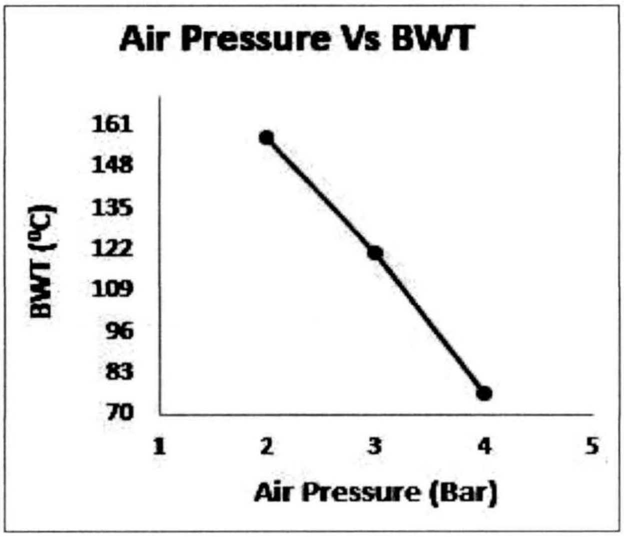

Fig. 10. Air pressure Vs BWT.

The experiments are conducted by varying the control parameters based on the Taguchi design of experiments and the responses are shown in Table 3.

6. Results and Discussion

6.1 Characteristic curves

The performance characteristics of the response DFT and BWT with respect to control factors are discussed in detail in the following figures.

From Fig. 7 it can be observed that the dry film thickness is increasing linearly from 0.571 mm to 0.933 mm by increasing air pressure from 2 to 3 bar and then increasing from 0.933 mm to 1.379 mm with increasing air pressure up to 4 bar.

From Fig. 8 it can be observed that the dry film thickness is increasing linearly from 0.571 mm to 0.646 mm by increasing spray time from 1.2 to 1.4 seconds and then increasing from 0.646 mm to 0.793 mm with increasing spray time up to 1.6 sec.

From Fig. 9 it can be observed that the dry film thickness is increasing linearly from 0.571 mm to 0.646 mm by increasing nozzle size (lock no.) from 20 to 26 and then increasing from 0.646 mm to 0.793 mm with increasing nozzle size up to 32.

From Fig. 10 it can be observed that the back-wall temperature is decreasing linearly from 157.2^{0}C to 121^{0}C by air pressure from 2 to 3bar and then decreasing from 121^{0}C to 76.4^{0}C with increasing air pressure up to 4 bar.

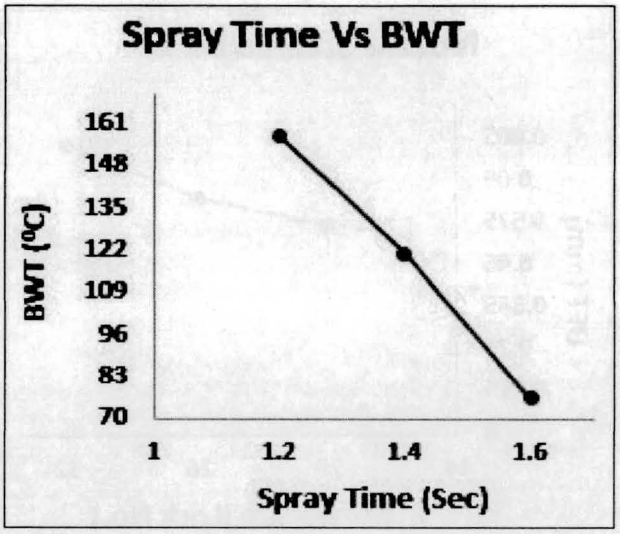

Fig. 11. Spray time Vs BWT.

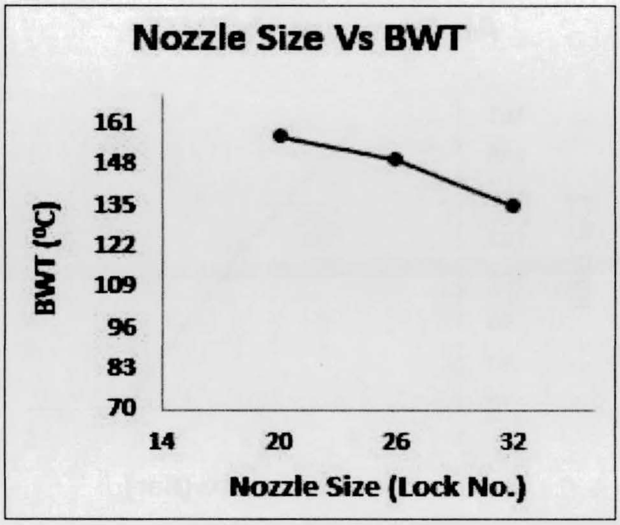

Fig. 12. Nozzle size Vs BWT.

From Fig. 11 it can be observed that the back-wall temperature is decreasing linearly from 157.2^{0}C to 113.2^{0}C by increasing spray time from 1.2 to 1.4 seconds and then decreasing from 113.2^{0}C to 88.7^{0}C with increasing spray time up to 1.6 sec.

From Fig. 12 it can be observed that the back-wall temperature is decreasing linearly from 157.2^{0}C to 149.7^{0}C by increasing nozzle size (lock no.) from 20 to 26 and then decreasing from 149.7^{0}C to 135^{0}C with increasing nozzle size up to 32.

6.2 Taguchi optimization technique

a) Dry Film Thickness (DFT)

The Main Effects plot for DFT and responses for are shown in Fig. 13 and Table 4 by using

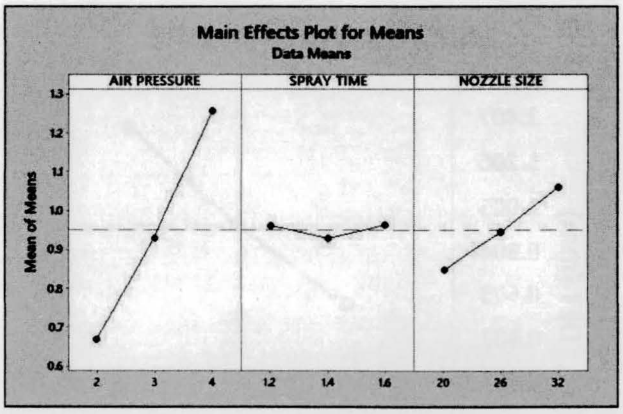

Fig. 13. Main effect plot for DFT.

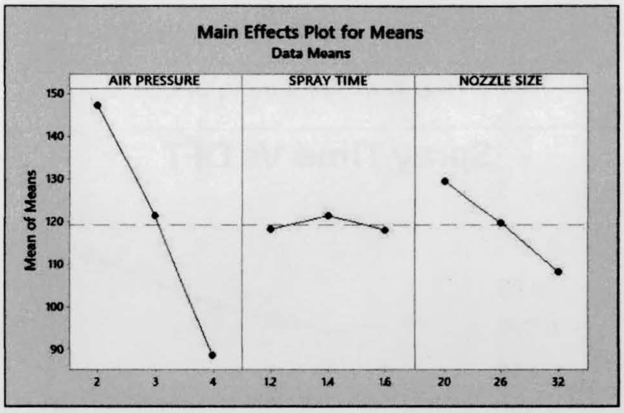

Fig. 14. Main effect plot for BWT.

Table 4
Responsesof means for DFT (Larger the better).

Levels	Air Pressure (Bar)	Spray Time (Sec)	Nozzle Size (Lock No.)
1	0.6700	0.9610	0.8477
2	0.9283	0.9293	0.9450
3	1.2553	0.9633	1.0610
Delta	0.5853	0.0340	0.2133

MINITAB R17 software gives optimal parameter combination i.e., air pressure 4 bar, spray time 1.6 sec and nozzle size 32 lock no.

b) Back-Wall Temperature (BWT)

The Main Effects plot for BWT and responses for are shown in Fig.14 and Table 5 by using MINITAB R17 software gives optimal parameter combination i.e., air pressure 4 bar, spray time 1.6 sec and nozzle size 32 lock no.

Table 5
Responses of means for BWT (Smaller the better).

Levels	Air Pressure (Bar)	Spray Time (Sec)	Nozzle Size (Lock No.)
1	147.30	118.20	129.53
2	121.47	121.37	119.80
3	88.77	117.97	108.20
Delta	58.53	3.40	21.33

Table 8
Confirmation test at optimal parameters.

Optimization Values Obtained from Taguchi Technique			Responses	
Air Pressure (Bar)	Spray Time (Sec)	Nozzle Size (Lock No.)	DFT (mm)	BWT (0C)
4	1.6	32	1.516	69.250

Table 6
ANOVA results for DFT.

Factors	DF	Seq SS	Adj SS	Adj MS	F-Value	P - Value	% of contribution
Air pressure	2	0.516280	0.516280	0.258140	3789.99	0.000	87.95
Spray time	2	0.002164	0.002164	0.001082	15.89	0.059	0.36
Nozzle size	2	0.068441	0.068441	0.034220	502.42	0.002	11.66
Error	2	0.000136	0.000136	0.000068			0.02
Total	8	0.587022					100

Table 7
ANOVA results for BWT.

Source	DF	Seq SS	Adj SS	Adj MS	F-Value	P-Value	% of contribution
Air pressure	2	5162.80	5162.80	2581.40	3789.99	0.000	87.95
Spray time	2	21.64	21.64	10.82	15.89	0.059	0.36
Nozzle size	2	684.41	684.41	342.20	502.42	0.002	11.66
Error	2	1.36	1.36	0.68			0.02
Total	8	5870.22					100

6.3 ANOVA

The Results of ANOVA and percentage of contribution of each parameter for DFT are shown in Table 6.

From the Table 6, the percentage of contribution of values for DFT it is observed that Air pressure (87.95%), spray time (0.36%), nozzle size (11.66%). It is observed that the air pressure has greater influence on DFT. Since this analysis is a parameter-based optimization design, from the above values it is clear that air pressure is the major factor to be selected to get the better DFT values. The back-wall temperature should be minimum for better performance of the thermal protection systems

The Results of ANOVA and percentage of contribution of each parameter for BWT are shown in Table 7.

From the Table 7, the % of contribution of values for BWT it is observed that air pressure (87.95%), spray time (0.36%), nozzle size (11.66%). It is observed that the air pressure has greater influence on BWT. Since this analysis is a parameter-based optimization design, from the above values it is clear that air pressure is the major factor to be selected to get the better BWT values.

6.4 Confirmation test

The confirmation test for the optimal parameters is conducted to evaluate quality characteristics for the coating process for LDAM. Table 8 shows the confirmation test results of the coating process.

7. Conclusions

The following conclusions are drawn from the present work:

- The Low-Density Ablative Material (LDAM) is prepared by using vinyl terminated silicone resin reinforced with hollow glass microspheres.
- The prepared LDAM material is sprayed by using High Velocity Low Pressure (HVLP) spray coating process for the application of thermal protection systems by varying the process parameters such as air pressure, spray time and nozzle size (lock no.).
- The optimum parameters obtained from Taguchi technique for DFT and BWT at air pressure 4 bar, spray time 1.6 sec and nozzle size 32 (lock no)
- Among the process parameters air pressure, nozzle size and spray time; air pressure is highly influential parameters followed by nozzle size and spray time.
- The optimum conditions for larger dry film thickness and smaller back-wall temperature, the LDAM material gives optimum results when compared to other composite materials.

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