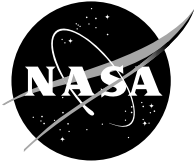# Optimal Substrate Preheating Model for Thermal Spray Deposition of Thermosets Onto Polymer Matrix Composites

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March 2003

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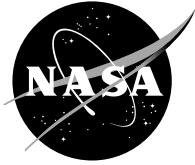
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Abstract

High velocity oxy-fuel (HVOF) sprayed, functionally graded polyimide/WC-Co composite coatings on polymer matrix composites (PMC's) are being investigated for applications in turbine engine technologies. This requires that the polyimide, used as the matrix material, be fully crosslinked during deposition in order to maximize its engineering properties. The rapid heating and cooling nature of the HVOF spray process and the high heat flux through the coating into the substrate typically do not allow sufficient time at temperature for curing of the thermoset. It was hypothesized that external substrate preheating might enhance the deposition behavior and curing reaction during the thermal spraying of polyimide thermosets. An additional difficulty arises from the low thermal conductivity and low specific heat capacity of the PMC substrate, which prevent effective substrate preheating by the HVOF jet as in the case of metallic substrates.

A simple analytical process model for the deposition of thermosetting polyimide onto polymer matrix composites by HVOF thermal spray technology has been developed. The model incorporates various heat transfer mechanisms and enables surface temperature profiles of the coating to be simulated, primarily as a function of substrate preheating temperature. Four cases were modeled: (i) no substrate preheating; (ii) substrates electrically preheated from the rear; (iii) substrates preheated by hot air from the front face; and (iv) substrates electrically preheated from the rear *and* by hot air from the front.

Thermal properties of the polyimide needed for the simulations were determined by Differential Scanning Calorimetry (DSC) and Thermo-Gravimetric Analysis (TGA). Microstructural characterization of the coatings and the morphology of polyimide splats sprayed both with and without substrate preheating were analyzed using standard metallographic techniques.

Coating temperature in cases (iii) and (iv) never dropped below the crosslinking temperature of the polyimide feedstock. This was the critical condition required for the curing reaction and successful deposition of thermosets by HVOF thermal spraying.

Introduction and Motivation

In response to the need for processable high temperature resistant polymer matrix materials, NASA developed a new class of thermosetting polymers known as PMR (Polymerization of Monomeric Reactants) polyimides [1]. Polyimide based matrix composites reinforced with carbon fibers are widely used in the aerospace industry due to their excellent high-temperature mechanical properties, thermo-oxidative stability and easy processability. PMR-15, for example, the first commercial generation of PMR resin, is being used to fabricate a range of aircraft engine components ranging from small, compression molded, bearings to large, structural autoclave-molded engine ducts used on the F404 engine for the US Navy FA-18 Hornet. A cost/benefit study indicated that significant cost and weight savings [2] could be achieved by replacing the titanium duct with a composite one.

New applications of PMC's in turbine engines, however, require improvements in composites' environmental durability, which may be achieved by the development of functionally graded material (FGM) coatings based on a PMR polyimide matrix, similar to the substrate matrix material, filled with varying volume fractions of ceramic particles. This requires that the polyimide matrix be fully crosslinked during deposition in order to maximize its engineering properties.

Thermal spray technology is being studied as an efficient tool for the deposition of polyimide based FGM coatings onto PMC substrates. Thermoset deposition by thermal spray technology is a challenge in itself due to the time sensitive nature of the *in situ* curing reaction of these polymers.

The limited work published on the HVOF spray deposition of polymers reports almost exclusively on the spraying of thermoplastics - Nylon, polypropylene, polycarbonate, PEEK, PAEK, PPS, PE, ETFE etc. Previous work on the plasma spraying of epoxy-based materials [3, 4] reported some difficulty in obtaining satisfactory build-up and properties of thermosetting polymeric coatings. This was attributed to the rapid heating and cooling nature of the thermal spray process and the high heat flux from the coating to the substrate, which did not allow sufficient time at temperature for curing of the thermoset to occur. The results indicated that external substrate preheating might enhance the deposition behavior and curing reaction during the thermal spraying of polyimide thermoset materials.

Preliminary proof-of-concept studies resulted in the successful spraying of pure polyimide and polyimide/WC-Co blends onto electrically preheated PMR-15 substrates [5, 6]. The substrate preheating setups were developed further during this work.

Problem Statement

The goal of this work was to determine temperature profiles of the thermosetting polyimide splats during HVOF spray deposition onto a PMC substrate. A temperature profile model of the coating was generated, primarily as a function of preheating temperature, by allowing for different heat transfer mechanisms. This model will help understand the process conditions necessary to produce highly crosslinked polyimide molecular architectures by the HVOF thermal spray process.

Background

The unit process in the deposition of thermally sprayed coatings is the impact of an individual droplet onto the substrate surface to form a splat. Assuming that a spherical droplet of diameter *d* is deformed into a disc or a cylinder of diameter *D* and thickness *h* under constant volume conditions, the thickness of an individual splat can be calculated using experimental values for the D/d ratio according to equation 1.

$$\pi d^3/6 = \pi D^2 h/4 \qquad (1)$$

For an average 50 µm diameter droplet, the calculated splat thickness would be ~8 µm. During cooling, the splat may undergo a change of state from liquid to crosslinked solid with the resulting enthalpy change acting as a heat source. Coating change of state can be incorporated into the heat transfer equations through the mean specific heat of the splat, calculated according to equation 2 [3].

$$C_p = [(C_{pc(Tx - \varepsilon)} + C_{pc(Tx + \varepsilon)})/2] + [\Delta H_x/(T_{x+\varepsilon} - T_{x-\varepsilon})] \qquad (2)$$

Where ΔH_x is the enthalpy of crosslinking, which relates to the area of the valley above the baseline on the DSC graph (Fig. 1, b-c). $C_{pc(Tx-\varepsilon)}$ and $C_{pc(Tx+\varepsilon)}$ are the specific heats of the lower

and upper limits of the liquid range, respectively. 2ε is the magnitude (in Kelvin) of the crosslinking temperature range of the polymer. The mean specific heat calculated in this way was 1,300 J/kg K.

Differential Scanning Calorimetry (DSC) and Thermo-Gravimetric Analysis (TGA) scans for the polyimide material are shown on Figs. 1 and 2, respectively. Numerical values obtained from these plots and reported thermal properties [1, 7] of the PMR polyimide thermoset material are listed in Table 1.
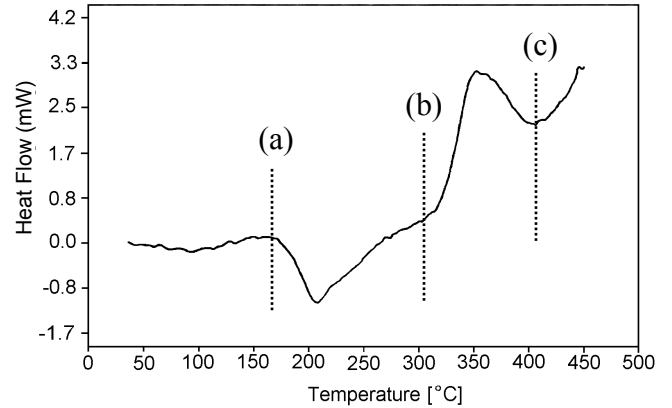

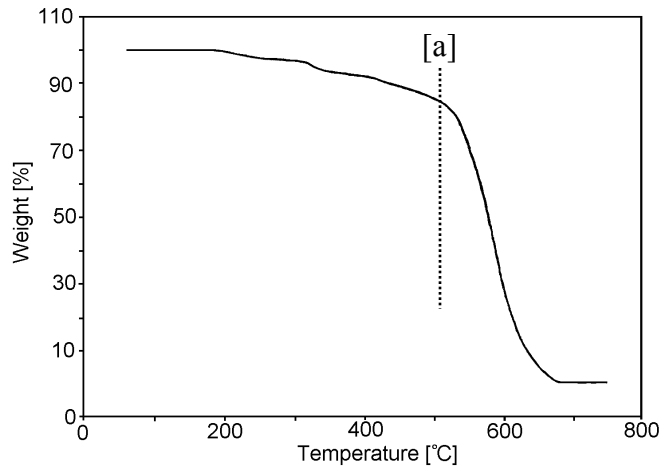*Figure 1: DSC (in air) scan result for the polyimide powder.*

Figure 2: TGA (in Air) profile of the polyimide powder.

Table 1: Thermal properties of the PMR polyimide powder.

Relaxation/Fusion Temp. (°C)	170 - 250
Crosslinking Temperature (°C)	300 - 400
Degradation Temperature (°C)	≥ 500
Specific Heat (J/kg K)	1,300
Thermal Conductivity (W/m K)	0.3
Density (kg/m³)	1,400
Gel Time @ 300 °C (s)	~15

The thermal conductivity of composite materials can vary widely based upon both fiber orientation and volume fraction. Investigations by several researchers [7] indicated that the thermal conductivity of graphite epoxy composites can be as low as 1.0 W/m K. A thermal conductivity of 1.0 W/mK and specific heat of 900 J/kgK were assumed for the graphite fiber-reinforced polyimide composite used in this work.

The maximum allowed substrate preheating temperature of 320 °C was specified as a temperature at least 28 °C below the glass transition temperature (T_g) of the fully cured substrate matrix polyimide (T_g = 350 °C), as recommended [8]. The maximum temperature of the HVOF combustion spray jet is ~3,500 °C and thus the outermost layers of thermoset particles may be continuously decomposing during their flight to the substrate. The mean temperature of the particle immediately prior to impact will thus be between its softening and decomposition temperatures, and calculations indicated that it was closer to the latter. For the purpose of these calculations, the temperature of the droplet immediately before impact was equated with the decomposition temperature of the polymer (500 °C), since this provided a definite value (Fig. 2).

Experimental Approach

In order to validate the theoretical model, a pure polyimide coating was sprayed onto a PMC substrate, preheated according to theoretical model, using a Stellite Coatings, Inc. Jet Kote® II HVOF combustion spray system. The coating was sprayed using the spray parameters summarized in Table 2.

Table 2: Typical HVOF spray parameters for spraying pure polyimide onto graphite fiber reinforced PMR-15 substrates.

HVOF Process Parameter	Value
Spray Distance (m)	0.15
Polyimide Feed Rate (gm/min)	2 - 9
WC-Co Feed Rate (gm/min)	20 - 50
Carrier Gas	Ar
Carrier Gas Flow Rate (m^3/s)	0.5×10^{-4}
H_2:O_2 Ratio	0.4- 0.5
H_2 and O_2 Flow Rates (m^3/s)	3×10^{-3}/6-7 $\times 10^{-3}$
Surface Speed (m/s)	0.11
Substrate Temperature (°C)	280 - 340

A molding grade of thermosetting polyimide (produced by Maverick Corp.) in an oligomeric form was mechanically crushed and cryo-ground (Shamrock) to produce a flowable (–100, +20 µm) particle size range powder suitable for HVOF spraying. The substrates used were 25 x 75 x 3 mm (1 x 3 x 1/8 in.) coupons of carbon-fiber reinforced PMR-15 polymer matrix composite material. Glass slides were used to observe individual polyimide splats. Coating microstructures and splat morphologies of polyimide sprayed both with, and without, substrate preheating were analyzed using standard metallographic techniques.

Omega type PT 502/120 electric strip heaters were used as an external heat source from the rear, together with hot air preheating from the front face of the substrate, as described later in Case 4.

Simulation Details

To a first order approximation, the heat flow in the radial direction of the coating may be neglected and the cooling of a splat can be simplified to a one-dimensional non-steady-state heat flow through the splat thickness to the substrate. This enables the conductive heat flow to be described by the principal equation:

$$(k/\rho C_p) (\partial^2 T/\partial x^2) = (\partial T/\partial t) \qquad (3)$$

where T is the absolute temperature within the splat at a distance **x** from the splat/substrate interface, **t** is the time and **k, ρ** and C_p are the thermal conductivity, density and specific heat of the splat.

A finite-difference method was used to solve the above equation, which in explicit form can be written as:

$$k_s(T_{i+1,j}-2T_{i,j}+T_{i-1,j})/\Delta x = (\rho_s C_{ps}\Delta x/2)(T_{i,j+1}-T_{i,j})/\Delta t \qquad (4)$$

Since temperature $T_{(x, t)}$ is a function of both time and distance, in order to solve equation 4, discretization must be conducted along two axes – **t** and **x**, (Fig. 3).

If the temperatures $T_{i+1, j}$, $T_{i, j}$ and $T_{i-1, j}$ are known then temperature $T_{i, j+1}$ can be determined, as shown in Fig. 3.
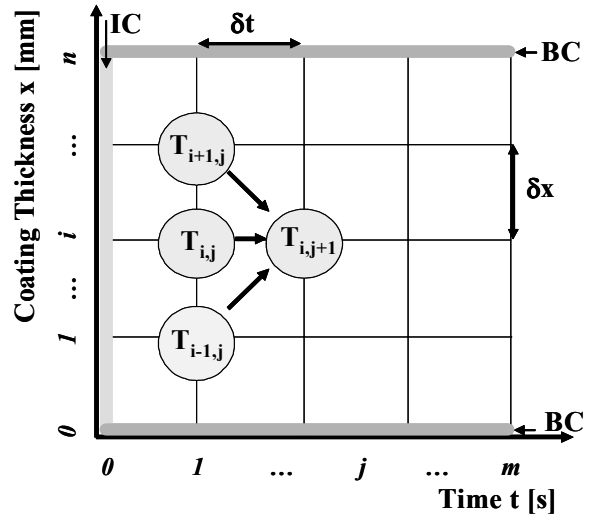

Figure 3: Schematic of the discretization model along two axes – x and t. BC = boundary conditions, IC = initial conditions.

The finite difference equations for the five boundary conditions (BC) shown in Fig. 4 were:

- At the substrate/environment interface:

$$k_s(T_b-T_a)/\Delta y + h_E(T_E-T_a) = (\rho_s C_{ps}\Delta y/2)(T'_a-T_a)/\Delta t \quad (5)$$

- Within the substrate:

$$k_s(T_h-2T_g+T_f)/\Delta y = (\rho_s C_{ps}\Delta y/2)(T'_g-T_g)/\Delta t \quad (6)$$

- At the substrate/coating interface:

$$k_c(T_l-T_k)/\Delta x + k_s(T_j-T_k)/\Delta y$$
$$= (\rho_s C_{ps}\Delta y/2)(\rho_c C_{pc}\Delta x/2)(T'_k-T_k)/\Delta t \quad (7)$$

- Within the coating:

$$k_c(T_r-2T_q+T_p)/\Delta x = (\rho_c Cp_c\Delta x/2)(T'_q-T_q)/\Delta t \quad (8)$$

- At the coating/environment interface:

$$k_c(T_u-T_v)/\Delta x + h_E(T_E-T_v) = (\rho_c C_{pc}\Delta x/2)(T'_v-T_v)/\Delta t \quad (9)$$
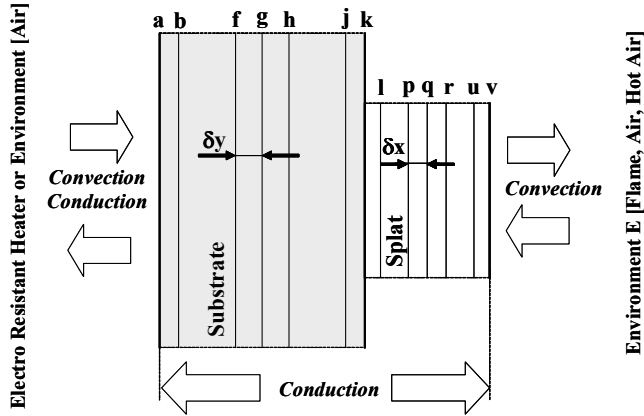

Figure 4: Schematic of differential elements used for the heat transfer analysis.

The required initial (IC) condition (equation 10) is of the form:

$$T_{(x,0)} = T_{in} \quad (10)$$

The following four cases were modeled, assuming different initial substrate temperatures:
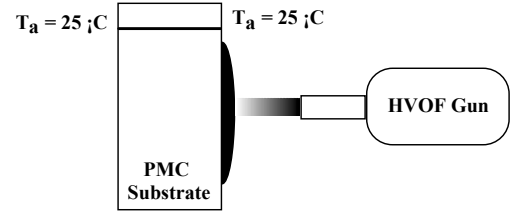
- **Case 1**: No substrate preheating (Fig. 5).

Figure 5: Schematic of the substrate/gun system with no substrate preheating.

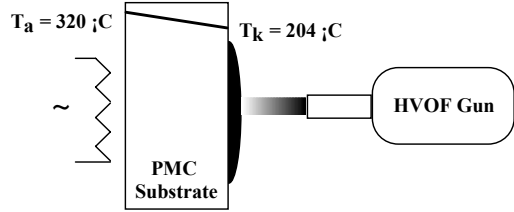- **Case 2**: Substrate electrically preheated from the rear (Fig. 6).

Figure 6: Schematic of the substrate/gun system with the substrate preheated from the rear only.

- **Case 3**: Substrate preheated by hot air from the front face (Fig. 7).
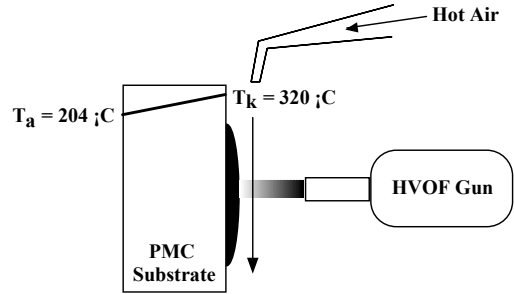

Figure 7: Schematic of the substrate/gun arrangement with the substrate preheated from the front face only.

- **Case 4**: Substrate preheated by the electrical resistance heater from the rear and by hot air from the front side (Fig. 8).
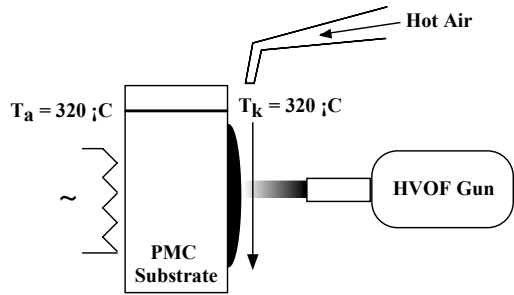

Figure 8: Schematic of the substrate/gun system with the substrate preheated from the rear and front faces.

In all cases the initial linear temperature gradient along the substrate cross-section was calculated using equation 11:

$$k_s = (T_a - T_k)/L = -q \qquad (11)$$

where k_s is the thermal conductivity of the substrate, T_a and T_k the temperatures of both surfaces of the substrate, L the thickness of the substrate and q the heat flux generated by the heaters. The time interval Δt was governed by the stability condition (equation 12) using a non steady-state finite difference method.

$$(k/\rho C_p)(\Delta t/\Delta x^2) < 1/2 \qquad (12)$$

Which gave $\Delta t = 2 \times 10^{-6}$ s. The substrate thickness (3 mm) was discretized using 5 equi-spaced points; and each 8 μm thick splat was divided into 3 equi-spaced points. The temperature within each layer (splat) was calculated until a steady-state temperature condition was achieved. Once this equilibrium was reached the deposition of the next layer (splat) was simulated.

Results and Discussion
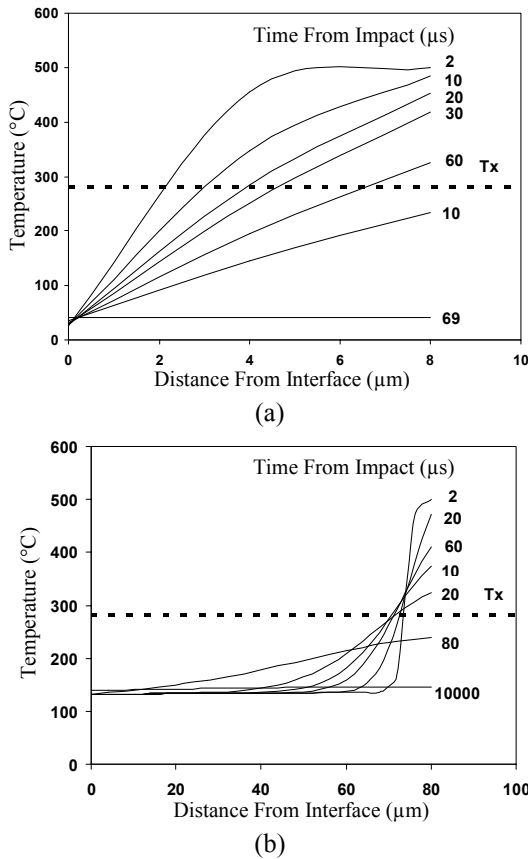
CASE 1: No substrate preheating.

(a)

(b)

Figure 9: Temperature profiles in polyimide coatings at various times after impact with the substrate: (a) single 8 μm splat; (b) 80 μm thick coating after deposition of the final (10th) splat.

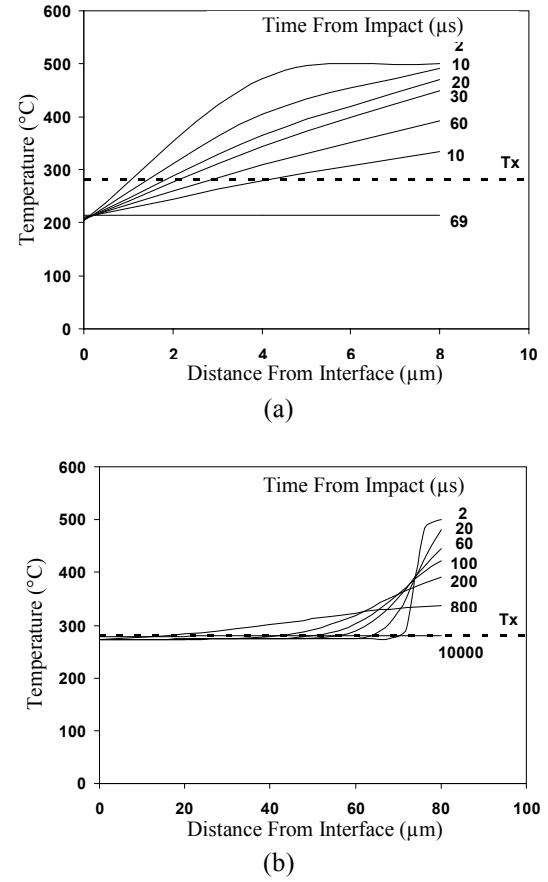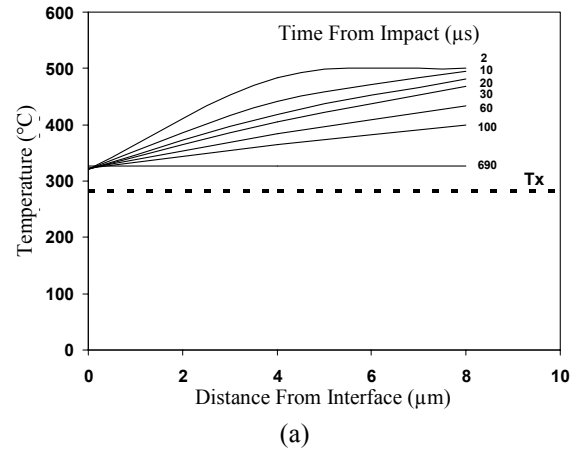**CASE 2:** Substrate electrically preheated from the rear.

(a)

(b)

Figure 10: Temperature profiles in polyimide coatings at various times after impact with substrate, (a) single 8 μm splat, (b) 80 μm thick coating after deposition of the final (10th) splat.

CASE 3: Substrate preheated by hot air on the front face only.
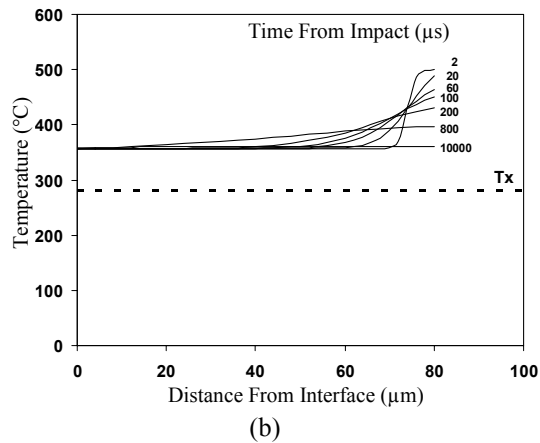

(a)

(b)

Figure 11. Temperature profiles in polyimide coatings at various times after impact with the substrate: (a) single 8 μm splat: (b) 80 μm thick coating after deposition of the final (10th) splat.

CASE 4: Substrate preheated by electrical resistance heater from the rear and by hot air from the front face.
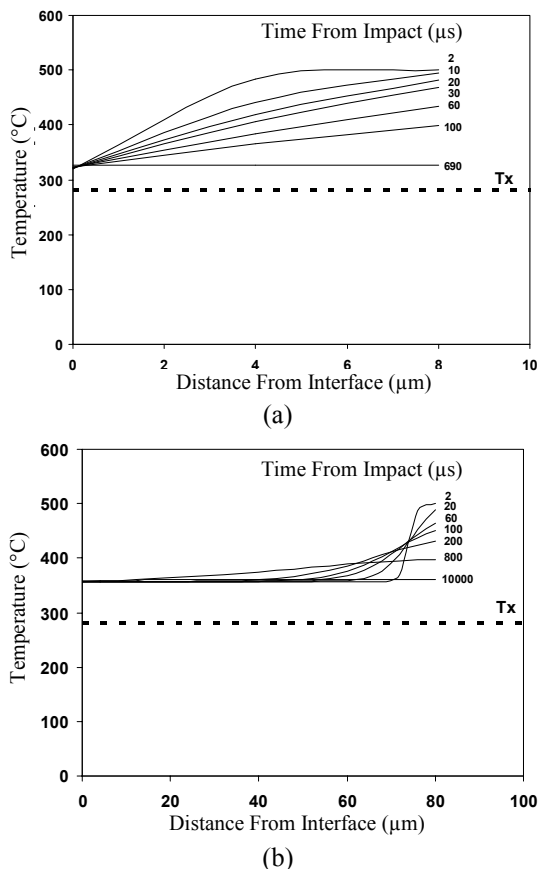

(a)

(b)

Figure 12: Temperature profiles in polyimide coatings at various times after impact with the substrate: (a) 8 μm single splat: (b) 80 μm thick coating after deposition of the final (10th) splat.

A theoretical model was developed to calculate the temperature profiles of 8 μm thick individual splats and 80 μm thick coatings built up from ten overlapping splats. In addition, temperature profiles were also calculated for the entire coating during the total time taken for the deposition of 10 individual layers. All calculations were done for four cases of substrate preheating. The temperature within each layer (splat) was calculated until a steady-state temperature was achieved. Once this equilibrium was reached the deposition of the next layer (splat) was simulated. Thermosets generally cure over a range of temperature, but for the purposes of this investigation, the crosslinking temperature (the horizontal dashed line shown on each graph at $T_x = 280$ °C) was taken as the lower limit of the range.

Case 1:
After a single droplet was deposited, its temperature decayed rapidly with time and all parts of the splat cooled from the initial 500 °C below the crosslinking temperature $T_x = 280$ °C in only 80 μs (Fig. 9a). The gel time of the polyimide was ~15 s @ 280 °C and hence the process model predicted that curing of an isolated 8 μm thick splat was not possible in cases where the substrate was not preheated. As the coating thickness increased with the deposition of successive layers, the time required to achieve the steady-state temperature increased to 500 μs for the 10th layer (Figs. 9b and 13) which was still several orders of magnitude shorter than the gel time.

Case 2:
The substrate was preheated to a maximum permitted temperature of 320 °C from the rear, but due to the low thermal conductivity of the PMC substrate, the temperature on the front side where the coating was being sprayed only reached 204 °C. This situation was slightly better than Case 1, since the temperature decayed rapidly below the crosslinking temperature in 300 μs. for one splat (Fig. 10a). It is interesting to note that after deposition of the 10th layer, the equilibrium coating temperature finally reached the polyimide crosslinking temperature (Figs. 10b and 14), which indicated that succeeding layers would remain above the crosslinking temperature.

Cases 3 and 4:
The coating temperature in cases 3 and 4 never dropped below the substrate/coating interface temperature (the baseline on Figs. 11a, b, 15, 12a, b and 16) thus in these cases the deposited material was always above the crosslinking temperature. This was the critical condition necessary for successful deposition of thermosets by HVOF combustion spraying. Further work will be carried out using Dynamic Mechanical Analysis (DMA) of coatings sprayed according to theoretical prediction in order to determine the crosslinking density of the polyimide coatings.
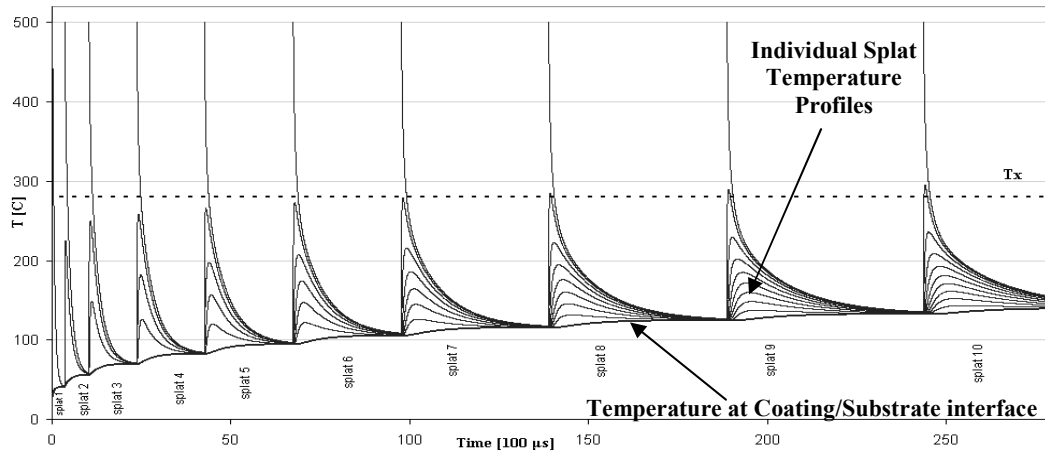
Figure 13: Temperature profiles for a ten-layer coating during deposition (~80 μm thick coating) – Case 1.

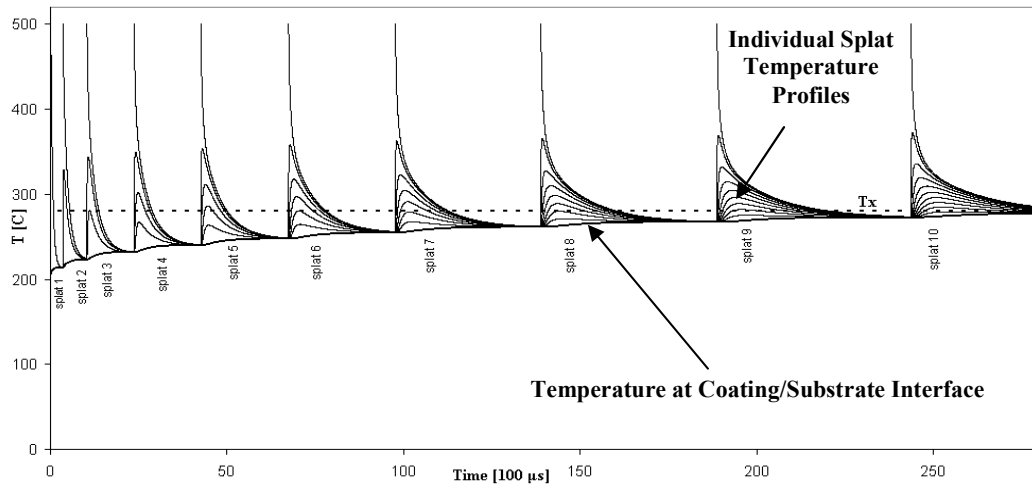*Figure 14: Temperature profiles for a ten-layer coating during deposition (~80 μm thick coating) – Case 2.*

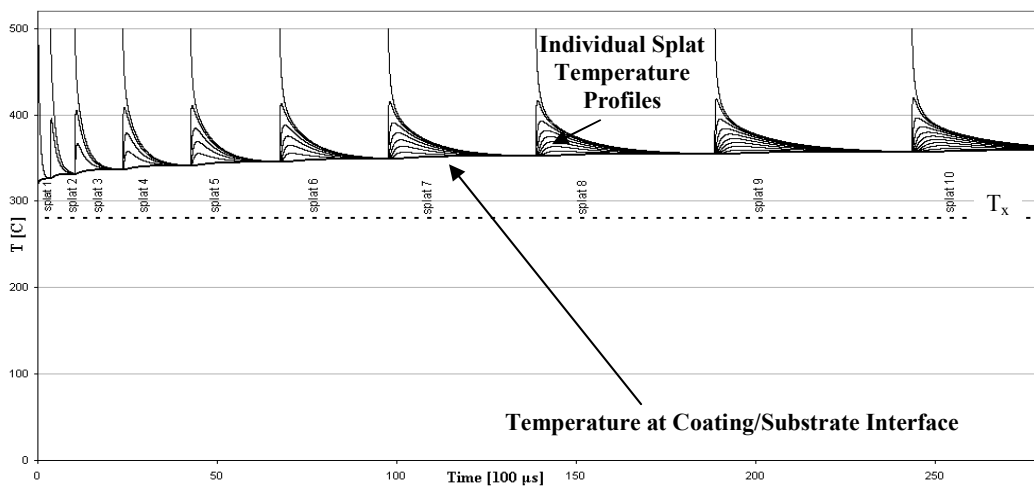*Figure 15: Temperature profiles for a ten-layer coating during deposition (~80 μm thick coating) – Case 3.*
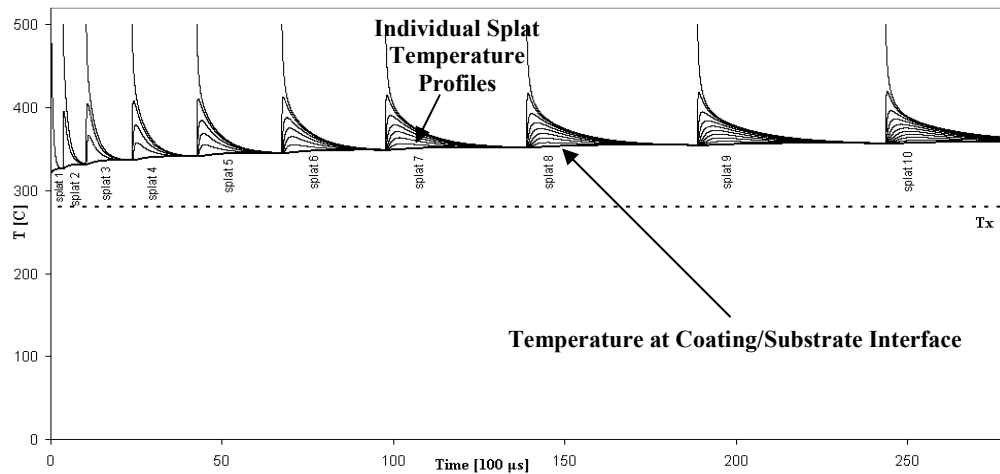
Figure 16: Temperature profiles for a ten-layer coating during deposition (~80 µm thick coating) – Case 4.

Polyimide splats sprayed onto glass slides with, and without, substrate preheating, are shown in Figs. 17 and 18, respectively.
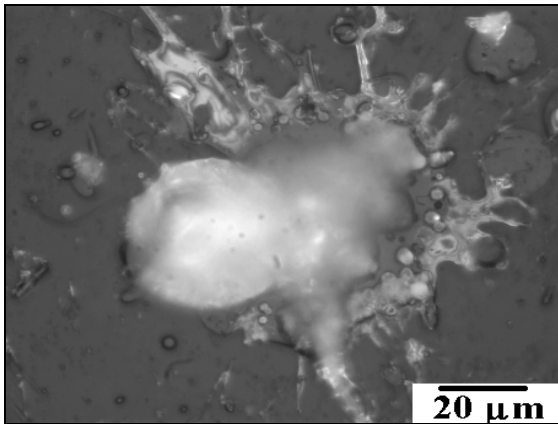

Figure 17: Partially melted polyimide splat on a glass slide.
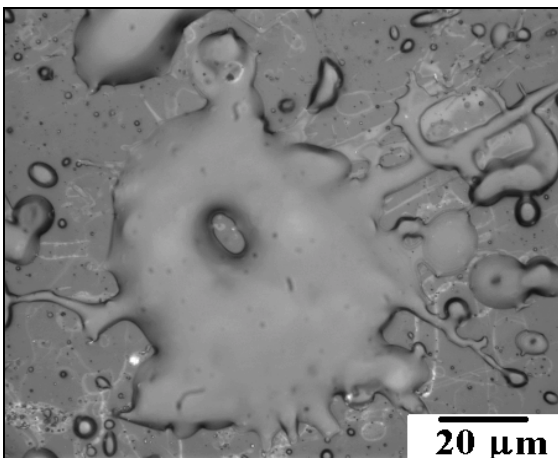
Figure 18: Well melted polyimide splat on a glass slide.

Splat tests indicated that external substrate preheating should enhance the deposition behavior during the thermal spraying of polyimide thermoset materials. Polyimide sprayed onto a preheated substrate resulted in well-melted splats (Fig. 18).

After the above tests, thermosetting polyimide coatings were successfully sprayed onto carbon-fiber reinforced PMC substrates preheated according to the theoretical model described in Case 4. Adherent polyimide coatings were obtained with little or no debonding at the coating/substrate interface (Fig. 19).

Figure 19: Pure polyimide coating sprayed onto a PMC substrate.

Conclusions

- A finite difference method has been demonstrated to be a viable tool for modeling the temperature profiles of thermosetting polyimide coatings deposited by the HVOF process.
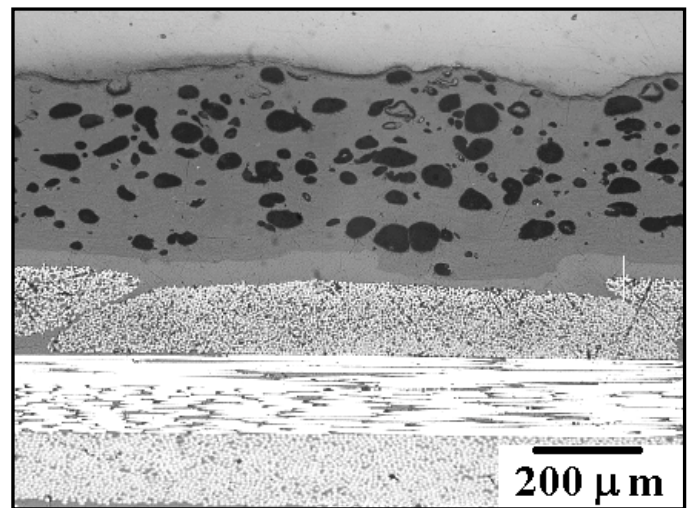
- The process model (Case 1) with no substrate preheating predicted that curing of isolated splats or a 10-layer coating could not occur.

- The theoretical model indicated that deposition of thermosetting polyimide coatings could be successfully achieved using the substrate preheating conditions described in Cases 3 and 4.

- Substrate preheating from the front using hot air (Case 3) was the recommended method since it was simpler than the dual preheating model (Case 4) and still maintained the required temperature profile within the coating.

- The finite difference method used was found to be very sensitive to the stability conditions. Modeling of the heat transfer through cross-sections of 8 μm thick splats required that the time interval be at the microsecond level ($\Delta t = 2 \times 10^{-6}$ s) in order to achieve stable results.

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High velocity oxy-fuel (HVOF) sprayed, functionally graded polyimide/WC-Co composite coatings on polymer matrix composites (PMC's) are being investigated for applications in turbine engine technologies. This requires that the polyimide, used as the matrix material, be fully crosslinked during deposition in order to maximize its engineering properties. The rapid heating and cooling nature of the HVOF spray process and the high heat flux through the coating into the substrate typically do not allow sufficient time at temperature for curing of the thermoset. It was hypothesized that external substrate preheating might enhance the deposition behavior and curing reaction during the thermal spraying of polyimide thermosets. A simple analytical process model for the deposition of thermosetting polyimide onto polymer matrix composites by HVOF thermal spray technology has been developed. The model incorporates various heat transfer mechanisms and enables surface temperature profiles of the coating to be simulated, primarily as a function of substrate preheating temperature. Four cases were modeled: (i) no substrate preheating; (ii) substrates electrically preheated from the rear; (iii) substrates preheated by hot air from the front face; and (iv) substrates electrically preheated from the rear and by hot air from the front.

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