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On the Reproducibility of Air Plasma Spray Process and Control of Particle State

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Controlling particle state is important to not only achieve the required microstructure and properties in coatings but also to clearly isolate and understand the role of other clusters of variables (such as the various substrate and deposition conditions) on the aforementioned attributes. This is important to design coatings for high performance applications and in the ongoing efforts toward achieving prime reliance. This study examines the variability in particle state and explores a few strategies to control them for improved reproducibility with the aid of in-flight particle and plume sensors. The particle state can be controlled by controlling the torch parameters or by directly controlling the particle state itself via feedback from particle and plume sensors such as DPV 2000 (Tecnar Automation Ltd, Quebec, Canada) and torch diagnostic system-spray plume trajectory sensor (TDS-SPT) (Inflight Ltd, Idaho Falls, ID). There exist at least a few control protocols to control the particle state (predominantly temperature and velocity) with judicious choice of critical parameters. In the present case particle state has been controlled by varying the critical torch parameters (primary gas flow and arc current) in a narrow range using 8% YSZ of angular morphology (fused and crushed) with 10-75 μm size distributions in conjunction with a N_2-H_2 laminar (nonswirl) plasma. Two important results emerge: (a) The particle state resulting from averaged individual particle measurements (DPV 2000) is surprisingly stable with variability in T < 1% and variability in V of <4%. Ensemble approaches yield a somewhat higher variability (5% in temperature). Despite this the variability in basic coating attributes such as a thickness and weight is surprisingly large. (b) Applying a much simpler control strategy to only control the particle injection and hence the particle trajectory results in reduced variability in coating attributes.

Keywords air plasma spray process, particle diagnostics, particle state control, process control, repeatability, reproducibility

1. Introduction

Plasma spray process has been in widespread use for more than half a century. In the past two decades these processes are being considered and used for high performance and critical applications such as prime reliant yttria stabilized zirconia (YSZ) thermal barrier coatings (TBC) on parts exposed to high temperatures. Primarily there are two issues associated with achieving the required coating microstructure and properties in coatings. They are: (a) identifying the process parameters that result in the required microstructure and properties and (b) achieving the same microstructure and properties repeatedly (process reproducibility).

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1.1 Process Reproducibility and Process Maps

Science based approaches such as the development of process maps are useful tools not only in robust process parameter selection, but also allow distilling critical parametric variables and their effects (Ref 1, 2). These maps can be extended to link particle states with deposit states and will eventually enable linking coating design with process/materials development. They further allow effective feedback control strategies based on multiple parameters. But they provide little or no information on the reproducibility of the process.

Process reproducibility is affected by the multitude of variables involved in the process (Ref 3). It is appreciated that some variables have profound influence on the reproducibility compared with the rest. Some are dynamic variables such as voltage fluctuations and anode (nozzle) wear (Ref 4). Therefore understanding the process reproducibility is a necessary step to the industry's ongoing efforts to achieve prime reliance. For prime reliance to be possible, the process must be controlled and made highly reproducible.

1.2 Particle State Control

It is known that both performance and properties of coatings are influenced largely by the microstructure, which in turn, is influenced by the particle state along with substrate and deposition conditions (Fig. 1). Hence controlling these clusters individually and varying them systematically is important to assess the role of each of the variable clusters and to enable tailoring the structure and properties of coatings.

Substrate and deposition conditions are relatively easier to control compared to the spray stream. Complex and dynamic

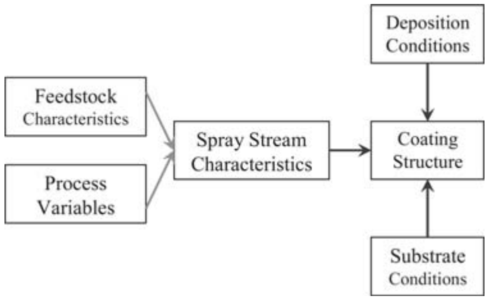

Fig. 1 Fundamental blocks in the APS process sequence

nature of spray stream makes it difficult to measure, assess, and control the particle state. Understandable yet uncontrollable drift in the process due to nozzle (anode) wear adds to the complexity. The advent of process diagnostics as well as models over the past decade has expanded our capability to critically examine the process state in light of process reproducibility (Ref 5-10).

1.3 Options to Control the Particle State

There exist at least a few different protocols to control the particle state (predominantly temperature and velocity) with judicious choice of critical parameters. Some are based on ensemble measurements and others on single particle measurements (Ref 5, 11-13). Data from single particle measurements can be used in two ways: (a) they can be averaged over a large number of particles to give good confidence on the data and (b) plotted as distributions to provide more information on the T and V. [Details on the significance of such distributions is reported elsewhere (Ref 14).] When it comes to control of the particle state in near-real-time averages are the appropriate choice, hence averages are used in the current study.

The particle state can be controlled directly or indirectly. Fixing the torch parameters and letting the process take its own course is the simplest method. It is also the principal method used for industrial coatings. The emergence of in-flight sensors now allows us to monitor the drift in the process but no control is exercised. This traditional method along with process monitoring can be referred to as the passive sensor method.

The other approach is directly controlling the particle state itself based on feedback from the sensors. T and V are appreciated to be good descriptors of the particle state although nondimensional and/or normalized parameters such as melting index and Reynolds number are perhaps appropriate (Ref 10). Due to the fact that most of the methodologies applied today rely on manipulating T and V and because those are the directly measured parameters, as a preliminary step we consider the same in our monitoring-feedback-control procedure. We refer to this approach as the active sensor method.

In this study, three significant torch parameters (independent variables), namely primary flow, secondary flow, and current, and only two particle properties (response parameters), were considered in this study. Thus the same average particle T and V could be obtained with multiple combinations of significantly different torch parameters. Manipulating all torch parameters to achieve the required T and V without setting limits can lead to drastic changes in the process resulting in complex interpretations.

Therefore in this study, a limited control envelope of the process space was considered to achieve the same average T and V. Based on the present understanding, it can be achieved by fixing one of the three torch parameters. Of the three significant torch parameters, primary flow strongly influences the average particle V whereas secondary flow and current strongly influence the average particle T. Thus primary gas flow was chosen as a control parameter (to vary V). It is known that varying hydrogen flow to achieve constant voltage/power could cause significant changes to the plasma, which could result in very different deposition efficiencies and coating properties (Ref 15). Hence arc current was chosen to be the other control parameter (to vary T). Thus, primary flow and arc current were varied in this study to achieve the required T and V while keeping secondary flow (hydrogen) constant.

An attempt is made to understand the reproducibility of the process from the point of view of particle state and also to identify methods that would improve the process reproducibility. We present some of the recent activities that shed new light on the relevance and implications of these strategies so that appropriate value judgments can be made by the industries.

2. Experimental Details

2.1 Processing and Diagnostics

Angular morphology (fused and crushed) 8% YSZ with 10-75 μm size distribution was chosen for the study. APS 7MB torch from Sulzer Metco with an 8 mm 'G' nozzle was used with nonswirl gas distribution. A mixture of nitrogen (N_2) and hydrogen (H_2) gas was used for plasma generation throughout this study.

Detailed diagnostics was performed using the different sensors that are set up in an integrated fashion at the Center for Thermal Spray Research (CTSR). Measurement of particle T and V using DPV 2000 (Tecnar Automation Ltd, Quebec, Canada) (Ref 16) on very large number of particles (10,000 typically) were averaged and was used for particle state control. Ensemble sensors such as the inflight particle pyrometer and spray plume trajectory sensor (IPP and SPT) (Ref 11) were also used. Schematic of the integrated sensor set-up and details about the sensors and the procedure followed can be found elsewhere (Ref 14).

Deposits were obtained on grit blasted 3 mm thick Al 6061 T6511 substrates of dimensions 228 × 25 mm at 130 mm standoff. The deposition procedure and all the related parameters were maintained the same for all the experiments. No specific effort was directed toward achieving a specific substrate temperature but because the experiments were in the similar operational parameter range and because the same procedure was followed, the substrate temperatures were within comparable range for all the experiments (~300 °C).

2.2 Passive Sensor Method

Medium values of the three torch parameters, namely nitrogen flow (N_2), hydrogen flow (H_2), and current (I) were selected for the study based on a design of experiment (DoE) exploring the hardware limits conducted earlier at CTSR (Ref 17) (47.6 SLM N_2, 5.6 SLM H_2 and 550A).

Six experiments, one every day for six consecutive days, were performed following the traditional method of setting the torch parameters and fixing the carrier gas flow. Instead of choosing some arbitrary value of carrier gas flow, injection was *optimized* (refer to sec. 2.3.1 in this article) for the first of the six experiments and then this optimized carrier gas flow was used for the rest of the five experiments. (Injection was not optimized for every experiment.) This resulted in an average particle temperature in the range of 2650 to 2670 °C.

2.3 Active Sensor Method

Though the concept of active sensor method is simple, the procedure is quite drawn-out and significant. It involves optimizing the particle injection and then tuning in the in-flight particle properties to the required value for each experiment.

The average particle T and V to be achieved were chosen as 2661 °C and 125 m/s. This particular combination of T and V was obtained from the first order process map using the medium parameters (47.6 SLM N_2, 5.6 SLM H_2, and 550A). Because it is extremely difficult to achieve the same exact combination, a narrow range of ±10 °C for T and ± 2 m/s for V was allowed. Active sensor method involves two steps.

2.3.1 Controlled Particle Injection. Particle injection is critical for orthogonal external injection of low density powders such as YSZ. Hence injection was optimized for every change in torch parameters. The plume angle (angle made by the particle trajectory with the torch axis) that resulted in maximum average particle T and V was controlled by adjusting the carrier gas flow. This was done to maintain comparable plume trajectory for the different process conditions and for the different experiments. Error/variation in the measured particle property due to differences in location of diagnostics in the plume cross section (for the different experiments) is thus overcome. A detailed article on optimizing injection is in preparation.

2.3.2 Achieving the Same Average T and V. Process maps are used to identify the torch parameters that result in a given particle T and V (Ref 18). But it is known that the in-flight particle characteristics are influenced by re-ignition and by nozzle life (wear) (Ref 4). Hence the calculated combination of torch parameters does not result in the exact predicted/required T and V. To address this issue, the torch parameters were tuned

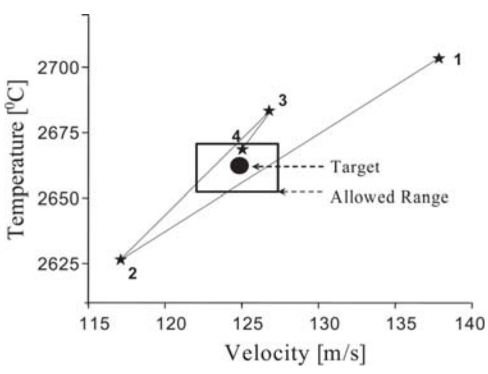

Fig. 2 Illustration of iterative tuning-in procedure

to achieve the same required T and V (target) for each experiment.

Tuning is done using the torch parameter vectors in T-V space following an iterative procedure (Fig. 2) to achieve the set stringent target of 2661 ± 10 °C and 125 m/s ± 2 m/s. Such a stringent target was met in four experiments using angular morphology YSZ. As mentioned earlier, the same average T and V can be achieved using multiple sets of torch parameters. However in the present case, the hydrogen flow (H_2) was maintained constant and the required T and V was achieved by adjusting the other two parameters (N_2 and I). This resulted in average particle temperatures between 2651 to 2668 °C in these experiments, just within the set limits.

3. Results and Discussion: Variability in Particle Characteristics and Coating Properties

3.1 Defining Variability and Setting a Standard for Comparison

Variability in the considered in-flight particle variable(s) and coating properties is calculated as the magnitude of difference between the extremes of each variable/property from the set of repeated experiments. This variability can be compared against the average obtained from the repeated experiments or to the maximum possible range of the variable for the given hardware. The range of values for each of the particle and plume related variables can be obtained from the first order process map linking the torch properties to the particle properties and plume characteristics (Ref 17). The same could be obtained for coating properties from a second order process map that relates salient process parameters to the coating structure and properties (Ref 1, 2). But due to the limitations in obtaining such data for the given set of hardware and feedstock, the variability is compared against the average value and is expressed in percentage.

$$\text{Variability in P} = \{\text{Pmax (n)} - \text{Pmin (n)}\}/\text{Average (n)}$$

where 'P' is the property under consideration and 'n' is the number of experiments.

3.2 Variables and Properties Used for Comparison

A few variables were selected to be compared between the two control strategies. For the in-flight particle properties, the average T and V obtained from single particle measurement of DPV 2000 is used. Data from at least 10,000 particles was collected and used. Ensemble properties from SPT and IPP such as the plume temperature and plume dip (angle made by the plume with the torch axis translated in terms of certain distance below the plume at the standoff) are also compared across the two methods.

From the coating perspective, simple coating characteristics such as coating thickness and weight are used to compare the effectiveness of both the methods. Volumetric porosity obtained by comparing the densities of the coating to the bulk (calculated by dimensional method) is also used for comparison.

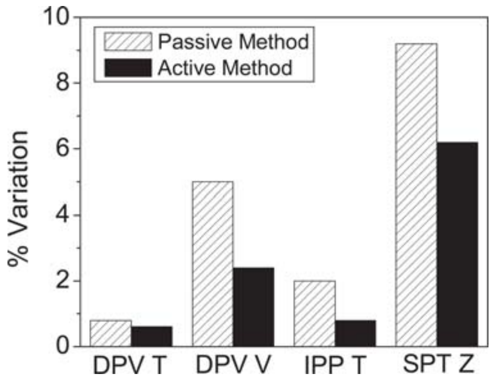

Fig. 3 Comparison of in-flight characteristics for passive and active methods

3.3 Comparing Passive and Active Sensor Methods

3.3.1 In-Flight Characteristics. The average particle properties (T and V) obtained from a few thousand particles measured at the plume flow center were analyzed. Surprisingly the variability in T was very small (compared to its average at a few thousand °C) for both passive and active sensor methods at less than 1%, though active sensor method showed slight improvement. The variability in V was halved for active sensor method from 5% for passive sensor method (Fig. 3).

The ensemble T measured by IPP, which is generally considered to capture overall changes in the plume better than the single particle measurements, shows higher variability as compared with the single particle averaged T. The variability in ensemble T as well as plume center location (as measured by SPT) was reduced for the active method compared with the passive method.

3.3.2 Coating Characteristics. It can be seen (from Fig. 3 and 4) that the variability in observed coating thickness, weight and porosity is large considering the small variability in particle state. In-spite of this, active sensor method has resulted in almost threefold reduction in variability in coating attributes compared with the passive sensor method. This shows that the variability in deposit can be controlled well by closely controlling the process at the in-flight/spray stream stage.

3.4 Role of Injection Optimization

To understand the role of injection optimization on the particle state and deposit formation, a set of four experiments was conducted fixing the torch parameters the traditional way but optimizing the particle injection for every experiment (passive sensor method + injection optimization; no tuning of particle state). Though this resulted only in a very small reduction in variability in particle state, clear improvement could be observed in the variability in coating thickness (Fig. 5) with injection optimization.

This could be attributed to the optimized and controlled particle trajectories as shown by the variation in 'SPT Z' (plume dip or the angle made by the plume with the nozzle axis as measured by SPT) in Fig. 5, resulting in lesser variability in coating assemblage. Surprisingly this is not reflected in the particle properties in-flight. This could be due to either or both of the following:

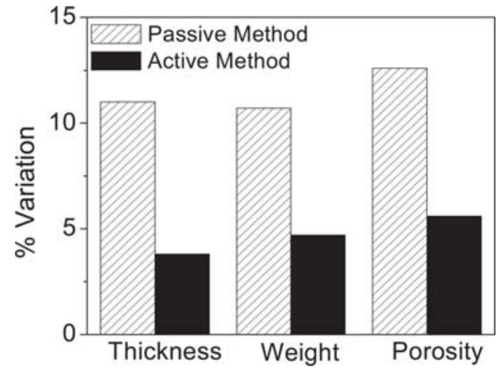

Fig. 4 Comparison of variability in coating properties for passive and active methods

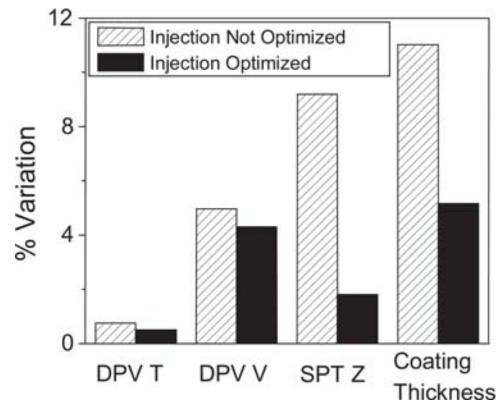

Fig. 5 Influence of injection optimization on the variability in particle and coating properties

- Low sensitivity of particle properties to small changes in particle trajectories around the optimum trajectory
- Insufficiency of in-flight diagnostics to capture all subtle changes in the process that influence the coating build-up

4. Summary and Conclusions

A systematic study was conducted to examine the variability in particle state and deposit properties from run to run for plasma spraying of YSZ. Three sets of experiments were conducted with integrated suite of diagnostics to monitor and in some cases control the process. The process was (a) only monitored, (b) monitored and injection controlled, and (c) monitored, injection controlled, and particle state controlled in a small operational window based on feedback from the sensors. Integration of various advanced diagnostic sensors allowed for assessment of variability in particle state.

Despite the small variability observed in particle state, substantial variability is observed in coating attributes. This calls for careful consideration of:

- measurement, analysis, and interpretation of the data
- the sufficiency of particle T and V to describe the particle state
- the stochastic nature of the coating build-up process, all of which could have resulted in the variability individually or combined

Feedback control based active sensor method appears to reduce the variability in in-flight characteristics as well as the coating properties considered herein. But it poses certain feasibility problems in large scale implementation. The choice of sensor, protocols for measurement and control, accuracy and sensitivity of the system, and the appropriate choice of control parameters would all require careful consideration. The detailed and meticulous steps involved in exercising control in real time such as establishing an injection optimization procedure, process map framework, and tuning-in procedure add to the issue. Furthermore the costs for such strategies would be significant and industry would need to make appropriate value judgment.

However, considering the simplicity of operation, passive sensor method along with injection optimization certainly offers ease of implementation and benefits with reduction in variability and is worth further consideration. Understanding of the spray stream characteristics and new strategies facilitated by sensor-based experimentation are necessary to further understand the outcome of this study.

Acknowledgments

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