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# In-flight Particle Diagnostics for On-line Process Control during Deposition of Plasma-Sprayed TBCs

C. Moreau, P. Gougeon, M. Prystay and L. Leblanc National Research Council Canada Industrial Materials Institute 75 de Mortagne Boucherville (Québcc) CANADA, J4B 6Y4

#### 1. SUMMARY

Plasma-sprayed TBCs are built by the successive addition of molten or partially-molten particles spreading upon impact on the substrate surface. Consequently, the temperature and velocity of the sprayed particles are among the most important parameters influencing the microstructure and properties of the deposited coatings. Online measurement of these parameters, as well as the particle trajectories, is thus an efficient way to monitor the state of the spray process. This approach should permits to minimize the influence of uncontrolled parameters such as the electrode wear or changes in particle injection conditions.

A rugged and easy-to-use optical sensor system has been developed to perform on-line particle diagnosis during spraying in a production environment. In this system, the thermal radiation emitted by the in-flight particles is collected by a compact sensing head and transmitted through an optical fiber bundle to a detection cabinet located away from the dusty environment around the plasma torch.

In this paper, a review of some applications of this diagnosis system used to determine the influence of the spray parameters on the particle temperature, velocity and trajectory and to control the spray process during deposition of TBCs are presented. The system was used in production at Pratt & Whitney Canada (PWC) for two months giving information about the stability and reproducibility of the plasma spray process.

#### 2. INTRODUCTION

Plasma spraying is a powerful technique that has been extensively used for the last twenty years to produce protective coatings on a large variety of substrates. During this period, the technique progressed mostly in an empirical manner, attention being focused on the effect of input spray parameters on the resulting coating properties [1,2]. Consequently, the spraying process control was implemented by monitoring and regulating the spray parameters to keep them at a predetermined optimum value. Arc current and power, arc gas flow rates, powder feed rate, powder carrier gas flow rate, net plasma energy are the main parameters usually monitored and automatically controlled during plasma spraying. This control approach has important drawbacks. Indeed, it is complex because a large number of input parameters must be monitored and controlled. It is also incomplete because some variables, such as the electrode wear state, cannot be monitored at all. Moreover, due to the huge temperature and velocity gradients in the plasma plume, a very small change in the plasma shape or in the injection geometry may have a significant effect on the particle trajectories and thus on their thermal histories and velocities. Significant variations in the coating properties may result from these uncontrolled perturbations.

Plasma-sprayed coatings are built by the successive addition of molten or partially-molten particles spreading upon impact on the substrate surface. Thus, the temperature and velocity of the sprayed particles immediately prior to their impact are among the most important parameters influencing the microstructure and properties of the deposited coatings [3]. On-line measurement of these parameters, as well as the particle trajectories, is thus an efficient diagnostic tool for characterizing the spray process. Since this approach permits to minimize the influence of uncontrollable parameters, one should expect a closer control of the spraying process leading to a better reproducibility of the coating properties.

Different techniques have been already developed to carry out both temperature and velocity measurements of plasmasprayed particles [4-10]. However, the required equipment is difficult to transfer to an industrial environment due to its complexity and the use of large optical components and fragile laser devices. Other approaches have been also used to provide information on the plasma spray process by monitoring only the particle velocity [11,12] or the thermal radiation from the hot particles [13-16].

Over the last 20 years, the aerospace industry has acquired a large experience in controlling plasma spraying process. Furthermore, the quality standards have led to the development of numerous recipes to control the process. These controls have shown their efficiency as the different coatings succeed in passing the corresponding standard tests required by the industry. Using particle monitoring systems in the aerospace industry can however be useful to evaluate the process stability and then the efficiency of the existing process control. Moreover, more efficient control approaches such as the one based on the measurement of the temperature and velocity of in-flight particles may be the only way to achieve higher quality coatings required in future applications.

This paper first describes the particle monitoring system developed at NRC. This system is dedicated to monitoring of in-flight particles in the laboratory and in production. In a second time, a review of a few applications of the monitoring system is presented. A focus is made on the influence of the temperature and velocity of yttria-stabilized zirconia (YSZ) particles on the coating structure and on the

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# **3. PARTICLE MONITORING SYSTEM**

The monitoring system, the DPV2000, is based on the detection and analysis of the thermal radiation emitted by the hot sprayed particles [17,18]. This system, commercialized by Tecnar Automation Ltée [19], consists of a sensor head located near the spray gun, a detection box linked to the sensor head by an optical fiber bundle and a computer (PC) for analysis and statistical computation (Fig. 1). The dimensions of the sensor head are 9 cm long and 3 cm diameter. When a hot particle passes in the measurement volume defined by optics, its image is formed on a two-slit optical mask fixed on the end of an optical fiber inside the sensor head. This event gives rise to a two-peak signal at the output of the photodetectors receiving the radiation through the optical fiber. Fig. 2 shows a schema of the sensor head and the corresponding fields of view of the detection optics. The distance between the slits as well as the magnification of the detection optics being known, the particle velocity can be computed from the time of flight of the particle image between the two slits. The particle temperature is obtained from two-color pyrometry by analyzing the signals from the two photodetectors receiving the radiation in two separate spectral ranges. Finally the particle diameter is computed from the absolute radiation intensity at one wavelength and the temperature of the particle.

Simultaneously to the measurements on the individual particles, the monitoring system measures also parameters of the particle jet considered as a whole. This information is useful to detect problems with the injection of the particles in the plasma jet. For that purpose, the radiation of the



**Figure 1** Schema of the optical system. The thermal radiation from the particles is received by the sensor head and the corresponding signals analyzed by the personal computer.





particle jet is collected and imaged on a row of optical fibers located in the sensor head near the fiber used for the measurement of the individual particle parameters. The height of the field of view seen by the optical fiber row is 4 cm. The optical fibers are coupled to a CCD linear camera located in the detection box. The parameters of the particle jet are computed from the analysis of the video signal of this CCD camera which is a representation of the spatial distribution of radiation in the particle jet. Three different parameters are obtained from this analysis: the position of the particle iet relative to the axis of the sensor head, the width of the particle jet and its total radiative intensity. This last parameter is very useful to detect variations in the powder feeding rate which are detected as time variations of the intensity. Measurements of individual particle parameters and particle jet parameters are made simultaneously and give complementary information on the spraying conditions.

### 4. RESULTS AND DISCUSSION

# 4.1 Monitoring of particle properties during coating production

The objective of this study was to follow, during a relatively long period of production, the evolution of the particle characteristics as measured with the DPV2000 monitoring system [20]. For that purpose, the procedure to produce coatings has been slightly modified. Usually, at beginning of spraying, the operator waits until the process is stable before moving the torch in front of the part to be coated. For this study, when the process became stable, the torch stayed in place near the sensor head to allow the measurement of particle parameters for one minute with the monitoring system. In the same way, once the part was coated, the process was not stopped immediately and the torch came back in front of the sensor head for another one-minute measurement. All the measurements were carried out at 63 mm from the torch which is the usual spray distance.

An example of results obtained in this way is illustrated in Figs. 3a and 3b. These figures show the evolution of the

particle temperature just before and after spraving a YSZ coating on a specific part. In Fig. 3a, the spraying process starts at 50 seconds and, before reaching the stability criteria at 150 seconds, a large increase of temperature is noted. After the one-minute stability period, around 210 seconds, the torch moves in front of the part for coating deposition and there are no more particles measured by the monitoring system. In the example of Fig. 3, the spraying process lasts about one hour, and then the torch comes back in front of the sensor head for one minute during which the process is stable. After this minute, at around 4300 seconds on Fig. 3b, the process is stopped according to the normal procedure of PWC. Results similar to those of Fig. 3 have been obtained for all the parts sprayed with YSZ powders. These results show that the temperature of the sprayed particles is stable when the PWC criteria of stability are reached.

From these results, the average values on the 60 seconds of stability can be computed for each measured parameter, i.e. the particle temperature, velocity and diameter. The average temperature of the YSZ powder sprayed with 3 guns used in production is shown in Fig. 4. These results are obtained during production of coatings with three sets of identical electrodes using the same spraying conditions. The abscissa of Fig. 4 is the number of hours of operation for each set. Measurements before (down triangles) and after (up triangles) coating deposition are represented at the same abscissa if they correspond to deposition on the same part.



**Figure 3** Example of particle temperature evolution during coating production with YSZ particles. (a) before deposition, (b) after deposition.



**Figure 4** Time evolution of YSZ particle temperature for 3 guns used in production :  $(\mathbf{\nabla})$  before and after  $(\mathbf{\Delta})$  deposition on different parts.

The number of points differs for each gun as the guns were used for spraying different powders and only the measurements on YSZ particles are shown. It is worth noting that there is a difference of typically 20°C between temperature measured before and after deposition on each part. Even if this difference is small, it is significant, indicating that the conditions of the particles have slightly changed during spraying.

The guns 1,2,3 have been changed after 46, 58 and 48 hours of spraying, respectively. For gun 3, the reason of change is not imposed by consideration of stability of the process but is due to a problem of waterproofness in the torch. With this gun, the process was considered stable according to the PWC criteria and also according to the measurements on the particles (Fig. 4). The situation is different for guns 1 and 2 as the temperature and also velocity of the particles (not shown) vary significantly during the use of these guns in production. It appears that these variations are often related to variations of voltage between electrodes which is one of the parameters recorded by the operators. In fact, when the voltage variations are too large, the usual procedure is to stop spraying as it is the case for gun 1 after 46 hours of operation. Consequently, the two triangles at 46 hours correspond to a beginning of spraying but no deposition was made as the voltage variation was too large. Even if gun 1 was used for coating deposition during 46 hours, Fig. 4 shows that the spraying conditions of the particles have constantly and significantly changed after 35 hours of operation. It is worth noting that the process was controlled by keeping the input power constant. So, as the electrodes become worn, the voltage tends to decrease and the controller reacts by increasing the current in the gun. In the case of gun 1, it clearly appears that such a correction was too large leading to a significant increase of the temperature of the sprayed particles. A corresponding increase in the velocity of the particles was also noted.

The gun 2 was changed because it reached 60 hours of spraying time. In such a case, changing the gun prevents eventual problems which can appear during production of coatings. After the 58 hours of operation of gun 2, no significant change was noted for the particle temperature. However, an increase of the particle temperature has been observed between 40 and 46 hours of gun operation but after 46 hours the conditions become again similar to what they were before 40 hours. This variation of temperature between 40 and 46 hours is correlated with a variation of the voltage between the electrodes but this change was considered too small to stop the process. It should be noted that during production with guns 1 and 2 particle temperatures have varied by 180°C and velocities by 30 m/s. This means that, in spite of the process control used to produce coatings, the particle spraying conditions can vary significantly.

The values for the average particle parameters for each gun are given in Table 1. The values for each parameter are average values obtained during the first hours of use of each gun, that means during the period of time they give relatively stable conditions for the particles. From Table 1, it appears that the different particle parameters are very similar for guns 1 and 3 except the difference of 50°C in temperature. The differences with gun 2 are larger as the temperature difference reaches 82°C. From Table 1, it is clear that in spite of the control established on the process, there are still significant differences in the particle characteristics. These differences can be explained by a variation in the way the particles are injected in the plasma jet. This is especially important if the plasma characteristics change as we can expect if the plasma gun is changed. Consequently, a control of the particle state appears more sensitive to variations in the process conditions than the current approach based on the control of plasma power.

It was observed that, in the case of YSZ particles, the spraying conditions are relatively stable during the stability periods (Fig. 3) even if the temperature can slightly vary during deposition. This is not true in the case of finer

 
 Table 1 Particle parameters measured during the first hours of operation for 3 guns used in production.

	GUN 1	GUN 2	GUN 3
Temperature (°C)	2687	2769	2734
Velocity (m/s)	156	161	152
Diameter (µm)	55	67	60
Flux (part./min.)	1065	1290	1123
Jet position (mm)	-0.9	0.0	-0.5
Jet width (mm)	4.7	4.6	4.7
Jet intensity (a.u.)	1.0	1.3	1.1

metallic (NiCoCrW) powders, 27  $\mu$ m average diameter. Figure 5 shows the evolution of the particle jet intensity before and after deposition. In this case, the process didn't reach a stable operation point even after 3 minutes of spraying when the torch normally moves to spray the part to be coated. Such a long time to obtain stable spray conditions is likely related to the feeding conditions of the fine powder into the plasma.



Figure 5 Time evolution of the particle jet intensity collected by the CCD camera.

# 4.2 Effect of the wear of the electrodes

In order to better understand the effect of the wear of the electrodes during spraying, a series of measurements of the particle characteristics was carried out in the laboratory [21]. A torch was mounted with a new set of electrodes and the state of the particles was monitored as a function of the spraying time. At regular intervals, YSZ coatings about 500 µm thick were sprayed.

#### 4.2.1 Two-Dimensional Profiles

Four 2-D profiles were measured after 1, 8, 22 and 37 hours of spraying. Mean temperature, velocity and diameter of the sprayed particles were monitored during the scans. These profiles were collected by moving the sensor head with a X-Y unit, scanning in this way the measurement volume of the DPV2000 across the particle jet at 65 mm from the gun. The number of detected events which is related to the particle flux at the measurement volume location was also counted for a fixed period of time (25 ms). This number can't be taken as the true particle flux but is qualitatively related to it in the sense that both follow the same trends.

Figure 6 represents the evolution of the mean particle temperature with the spraying time. The temperature distribution as well as the velocity distribution (not illustrated here) followed the same behavior during the experiment starting with their highest values at the beginning of the experiment (one hour of spraying) and decreasing continually until the end of the experiment (37 hours of spraying). A variation of more than 200 °C is observed between the temperature distributions measured at the beginning and at the end of the forty-hour experiment. During this time, the velocity distribution has varied by more than 30 m/s. It is worth noting that, in this series of measurements, the plasma current was kept constant at 800 A. The results show that even though the input spray parameters stayed the same, the particle state has significantly changed. Such temperature variations may lead to important coating microstructure and thermal property changes [22].

Figure 7 shows the evolution of the distribution of the number of detected events which is related to the particle flux. At the beginning of the experiment the distribution was not symmetric, being nearly 50% wider in the horizontal direction. However, the distribution tended to get more symmetric with increasing spraying time. It is also observed in Fig. 7 that the maximum number of events decreased with increasing spraying time suggesting that the more the plasma gun was used the more the hot particles were lost from the spray jet. This has also been observed recently with alumina [23].

#### 4.2.2 Coatings and Deposition Efficiency

Coatings were sprayed after 3, 10, 22 and 37 hours of spraying in order to evaluate the effects of the sprayed particle state on the microstructure of the deposited material through the long-term experiment. Image analysis showed that the total porosity was the same for all coatings, at about 16%.

After three hours of spraying, the deposition efficiency was 55% while at the end of the experiment (37 hours of spraying) it fell to 41% reflecting important changes in the state of the sprayed particles. Figure 8 shows the microstructure of the coatings deposited after 3, 10 and 37 hours of spraying. As seen in the figure, a clear evolution of the coating microstructure took place with increasing spraying time. At the beginning, after 3 hours of spraying, the deposited coating contained a relatively large amount of cracking. The cracking pattern, though still present, is not so apparent in the coating sprayed after 10 hours. The mean particle temperature was nearly the same for these two coatings whereas the particle velocity differed by nearly 30 m/s. Coatings sprayed after 22 (not shown in Figure 8) and 37 hours were very similar in their microstructure. However, in these coatings cracking patterns like those observed after 3 and 10 hours of spraying did not form. These microstructure differences can be attributed to the different particle state before their impact on the substrate. Indeed, in a previous communication [22] it has been demonstrated with a similar ceramic powder that a mean particle temperature variation of 200°C, such as the one measured in the present experiment between 3 and 37 hours of spraying, can result in a more than 10% thermal diffusivity variation as well as significant microstructure changes. It should be reminded that between 3 and 37 hours of spraying the gun power



Figure 6 Temperature distribution (°C) of the sprayed particles after different spraying times. The gun axis corresponds to X = 0 mm and Y = 0 mm.



Figure 7 Number of detected events (thousands) after different spraying times. The gun axis corresponds to X = 0 mm and Y = 0 mm.



after 10 hours of spraying



after 37 hours of spraying



Figure 8 Pictures of coatings deposited after 3, 10 and 37 hours of spraying. The coatings are about 580, 480 and 520  $\mu$ m thick, respectively. The number of vertical cracks decreases with the spraying time as electrode wear becomes significant.

varied by only 1 kW. It would have been difficult, if not impossible, to predict any microstructure changes from this power variation. Present results show that coatings prepared after different spraying times with the same input spray parameters can result in materials having significant differences in their microstructures and properties.

#### 4.2.3 Attempts to Recover Initial Particle State

After the forty-hour experiment, the electrodes were disassembled from the plasma gun. They were mounted again several months later when attempts were made to try to recover the initial particle parameters. In a first attempt, the gun current was set to 900 A. However, no significant changes in the mean particle temperature and velocity were observed neither when gun current was increased from 800 A to 900 A and to 1000 A, nor when the powder carrier gas flow rate was changed. Nevertheless, when hydrogen proportion was changed to 5% (as compared to 3.9% vol. during the forty-hour experiment) the mean particle temperature increased by about 150°C (from 3000°C to 3150°C) whereas the mean particle velocity increased by more than 15% (from 148 m/s to 175 m/s). Moreover when the total gas flow rate was increased from 52.0 l/min to 60.6 l/min, keeping the hydrogen proportion to 5% vol., the mean particle temperature increased from 3150°C to 3200°C while the mean particle velocity increased from 175 m/s to 190 m/s.). These velocity and temperature are similar to those measured after two hours of spraying. The measured gun power was 35 kW, which is about 5 kW higher than its value after two hours of spraying.

Using these spraying conditions, 2-D profiles of the particle jet were measured. They are rather similar to those measured after eight hours of spraying (see Figs. 6 and 7). The numbers of detected events are similar to those observed after 8 hours of spraying (Fig. 7). A coating as well as a deposition efficiency test were performed using the new input parameters. The deposition efficiency was measured to be 53% which is essentially the same as what had been measured after three hours of spraying (55%) and much higher than the 41% value measured after 37 hours of spraying. The microstructure of the coating prepared



Figure 9 Picture of a coating deposited with higher hydrogen proportion and gas flow rate. The coating is about 450  $\mu$ m thick.

with higher hydrogen proportion and gas flow rate is shown in Fig. 9. It can be seen from this figure that the cracking pattern is rather similar to that of the coating sprayed after 3 hours, as seen in Fig. 8.

So after a long spraying time (about 40 hours) by changing some input parameters it was possible to recover the sprayed particle parameters obtained after three hours of spraying and to recover the same type of coating structure observed before significant electrode wear took place.

# 4.3 Controlling the particle state and coating structure

In order to implement a feedback loop to control the state of the in-flight particles, it is necessary to know how the input spray conditions influence the state of the particles. It is also necessary to know how the temperature and velocity of the impinging particles affect the coating structure. For TBCs coatings, one key characteristic is the crack network within the coating. Indeed, it seems advantageous to maximize the number of cracks perpendicular to the substrate while minimizing the number of parallel cracks [24].

An example of how the arc current and total gas flow rate influence the state of YSZ particles is shown in Fig. 10 [22]. The powder used in this study was a fused and crushed YSZ powder (-45 +11 µm). After setting the plasma current and total gas flow rate, the powder carrier gas flow rate was adjusted to position the spray jet axis at a constant angle of about 3 degrees below the torch axis. As shown in the process control plot of Fig. 10, an increase in plasma current increases both the temperature and velocity of the particles whereas an increase in the gas flow rate increases their velocity but decreases their temperature. Subsequently, conditions for spraying particles at an exact temperature and velocity may be obtained by carefully regulating both the arc current and gas flow rates. In this way it is possible to regulate the spray process in order to spray coatings in a reproducible manner.

After having established the data shown in Fig. 10, it is possible to optimize the coating structure by varying independently the temperature and velocity of the sprayed



Figure 10 Process control plot showing the effect of the arc current and arc gas flow rates on the particle temperature and velocity. The arc gas is Ar with 33% He and the powder is YSZ (-44 +11  $\mu$ m).



Figure 11 Average temperature and velocity of the sprayed particles for coatings sprayed in the study of *Prystay et al.* 

particles instead of varying the input spray parameters. For example, in the work of *Prystay et al.* [22], the coatings were sprayed in the 7 conditions shown in Fig. 11. The velocity and temperature effects were decoupled by selecting operating parameters to generate data points that fall along a staircase pattern. For example, the influence of particle velocity at about 2995 °C is seen by comparing coatings 1 and 7. (The average temperature between the particle temperatures of samples 1 and 7 is used. Other values shown on the graph were determined in the same manner.) The effect of velocity at particle temperatures of 2840 °C is seen by comparing samples 3 and 4. The results of changing particle temperature for fixed particle velocities are obtained by comparing sample 4 with 5 (200 m/s) and sample 3 with 2 (250 m/s).

In this work the angular crack distribution was characterized by image analysis using a dedicated algorithm developed in our laboratory. This algorithm treats each pore or crack as an individual feature of equal weighting and no results regarding their width are included. The orientation of each feature is determined and the total length of the features (in pixels) as a function of their orientation is calculated.

An example of results is given in Fig. 12 where the crack distribution of coatings 4 and 5 are compared. Both coatings were sprayed at particle velocity of 250 m/s but with different particle temperatures. In this case, the increase in temperature has the effect of sharply decreasing the number of horizontal cracks (0 degree) and increasing the vertical cracking (at 90 degrees). In fact, coating sprayed with particles at 250 m/s and 3014 °C exhibits only a weak preferred angular crack orientation. In addition, although this coating is not the densest in this study, it is the coating with the highest thermal diffusivity [22]. This suggests that the significant reduction in horizontal cracking decreased the thermal insulating properties of the coating.

#### 5. CONCLUSION

Plasma-sprayed coatings are used in a wide range of new and demanding applications. As the quality requirements of



Figure 12 Angular crack distribution in coatings 4 and 5 sprayed with particles at 250 m/s and (●) 3014 °C and (■) 2850 °C, respectively.

the sprayed coatings increase, the process control becomes a key issue in order to produce, day after day, high quality coatings in a reproducible manner. Monitoring the temperature, velocity and trajectory of in-flight particles before their impact on the substrate appears as an efficient way to characterize the process and to perform feedback on the input spray parameters. Moreover, it is also an efficient tool to optimize the spraying conditions by varying parameters that have the most crucial effects on the coating properties.

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