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Plasma spray processes: diagnostics and control?*

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Abstract: The expenses related to the rejection and replacement of imperfect coatings can reach 15% of the production cost. Thus, for manufacturers, the reproducibility and reliability of plasma sprayed coatings are the main goals for respecting quality standards and decreasing production cost. The aim of this paper is a tentative to answer to the questions:

—Which diagnostics can be used within harsh conditions prevailing in spray-booths?

—Which close-loop control can be achieved between the on-line measured parameters and the spraying process macroscopic parameters?

INTRODUCTION

The reliability and reproducibility of plasma sprayed coatings are critical to decrease the total production costs and help coatings to be more and more used as design elements even in extreme service conditions. Many errors are responsible for a weak reliability [1]:

- Errors in the design of parts to be coated which, as in the other surface treatment processes, can be lessened by a better collaboration between design department and production workshop.
- Errors of operators and errors in pre- and post treatments which can be avoided by an improvement in education and training of employees.
- Instabilities in the spray process which can be partly overcome by the use of reliable spray equipment and an on-line control. But the implementation of the latter depends on the knowledge of the key parameters controlling coating properties.

The first part of this paper will review some techniques and instrumentation to control the DC plasma spray process in an industrial environment. The second part will underline the key points of the process according to the present knowledge and various problems related to particle behavior in the plasma jet and coating formation. Finally, the third part will present some possible solutions concerning plasma torches and process control by developing a database associated to an on-line control.

EXISTING CONTROL TECHNIQUES

Today plasma spray equipment are equipped with microprocessors in close-loop using:

- Input macroscopic parameters such as: arc current, flow rates of plasma forming gases, electrode cooling water mass flow rate.
- Output macroscopic parameters: voltage, temperature difference of electrode cooling water to calculate on-line the torch thermal efficiency and gas enthalpy, and keep the latter constant for

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example by increasing automatically the arc current or the hydrogen volume percentage when it decreases due to electrode wear.

- Powder feeder parameters such as powder carrier gas flow rate and powder mass flow rate, both of them being more and more controlled in real time to keep them constant;
- substrate parameters especially substrate temperature to monitor it by acting on the cooling air flow rate;
- parameters linked to robotization and automation of the relative movement torch/substrate

At last the microprocessor indicates when the scheduled replacement of electrodes and injector tips is achieved in order to anticipate wear disturbances and drift of coating quality.

It is clear that rapid progresses have been made over the last decade in diagnostics used to characterize plasma jets, plasma–particle interactions, and particle–substrate interactions.

Many laboratories [2–5], have carried out experimental works on plasma jets and particles in flight or upon flattening. These works have resulted in techniques allowing measurements of a single particle velocity, temperature and size under thermal plasma conditions. These techniques often based on the measurement of the thermal radiation emitted by the particles, are now commercialized by companies:

- The 'in flight' company sells a system developed by IDAHO National laboratory for measuring the mean surface temperature of particles and controlling their mean trajectory (from the radial distribution of hot particles) using a pyrometer and a CCD linear camera [6].
- TECNAR Automation commercializes the DPV 2000, developed by CNRC of Boucherville, which controls the velocity, size and temperature of each particle [7,8].
- Control Vision commercializes an imaging technique to visualize particles in the hottest zones of the plasma jet and measure their velocities. The particles are illuminated by a N2 laser flash [9].
- SRATONICS proposes an imaging two-color pyrometer, using a CCD camera and allowing temperature measurement from 600 to 2700 K with an accuracy in the order of 10%. The system provides two images simultaneously at short and long wavelengths [10].
- Tampere University has developed a commercial imaging technique to measure hot particle density, velocity, and temperature. This imaging technique seems to be the more appropriate and simplest technique for the future [11].

All these techniques are rather expensive and cannot equip each spray booth, especially in small thermal spray workshops. The University of Limoges is developing a very simple and low cost system using a common CCD camera fixed on the torch [12]. It controls the stability of the spray jet and mean particle trajectory coupled with the substrate temperature. Such a tool has proved to be very sensitive to any drift in powder injection conditions and torch working parameters. It controls also the substrate temperature. The latter is a very important spray parameter on which depends the coating microstructure. Its measurement is achieved by a pyrometer technique. However to follow temperature close to room temperature, requires the use of an IR detector very sensitive to molecular IR absorption and emission.

KEY POINTS ACCORDING TO THE PRESENT KNOWLEDGE

The first point is linked to particle injection. The particle injection parameters control to a large extent the particle trajectory, spray jet expansion and deposition efficiency. Figure 1 shows the effect of the argon carrier gas flow rate on the maximum of light emitted by the hot particles at 70 mm from the nozzle exit. This maximum is obtained for a carrier gas flow rate of about 4.2 s.l.m, corresponding to a trajectory situated at 8 mm from the torch axis. This optimum corresponds to the mean momentum of the particles equal to that of the plasma jet [13].

The mean trajectory of the particles is disturbed by instabilities linked to powder feeder (a few tenths or hundredths Hz) and plasma jet fluctuations (a few kHz) [14,15]. The continuous movement of the anode arc root (restrike mode with Ar-H_2 mixtures) results in arc voltage fluctuations (2 and 20 kHz) which causes variations of the length and diameter of the plasma jet. The pictures in Fig. 2 captured using a Control Vision system show the interactions of the fluctuating plasma jet with particle injection at the exit of a plasma torch. Under the spraying conditions of this study, the angle between mean particle

KEYS POINTS ACCORDING TO THE PRESENT KNOWLEDGE

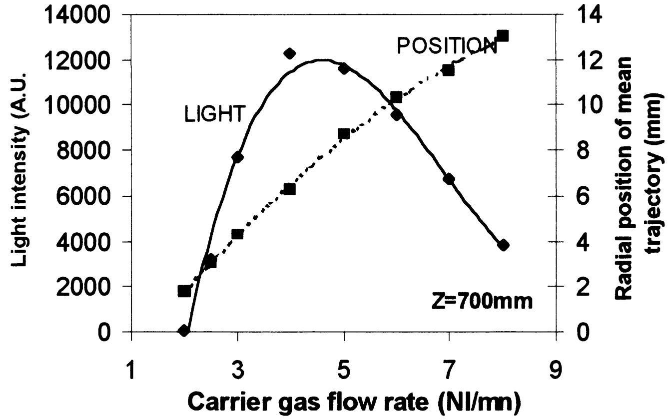

Fig. 1 Optimisation of carrier gas flow rate with measurements performed 70 mm downstream of the nozzle exit. (Alumina particles; –22 +45 μm; nozzle i.d. 7 mm; 500 A; 80 V; thermal efficiency 55%; 32/12 s.l.m. Ar/H_2; internal injection; injector i.d. 1.8 mm).

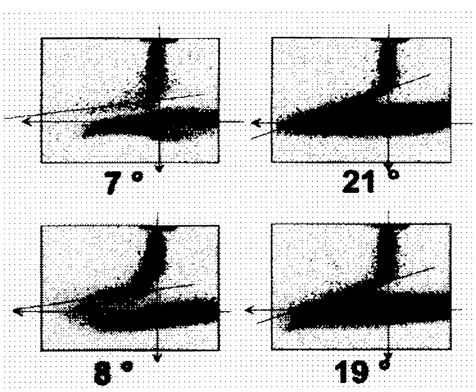

Fig. 2 Mo particles injected in a DC plasma jet.

trajectory and torch axis was between 7° and 20°. This is due to the plasma jet momentum variations with voltage fluctuations. [13]

The second point is related to particle evaporation. Often the particle surface evaporates before the particle core is melted (heat propagation phenomenon [16]), and in addition particles can react in flight with the plasma gas or surrounding atmosphere and undergo chemical reactions especially oxidation.

Figure 3 shows the effect of the hydrogen content in the plasma forming gas on the mass balance when spraying an iron powder. Depending on plasma parameters the vaporized mass fraction can reach 20% and more.

The mass loss at injection, estimated by comparing the profiles of cold and hot particles, varies from 40 to 30%. The total mass loss obtained from mass deposition efficiency increases from 40 to 60%. A rise in H_2 content results in an increase of the mass loss by evaporation and a decrease in the mass loss at the injection point because of an easier penetration of powder [12].

The other key points deal with the coating formation and particularly particle impacts [17], formation of resulting splats [18,19], and the layering of splats and passes. Using a laser long-distance microscope developed in our laboratory and devoted to the measurement of particle parameters just before impact, it is possible to observe impact phenomena with three different modes: rebound, deposition and splashing. Figure 4 shows the impact of alumina droplets with a mean velocity of about 100 m/s and a mean temperature of about 3000 K on a hot substrate ($T \approx 1500$ K).

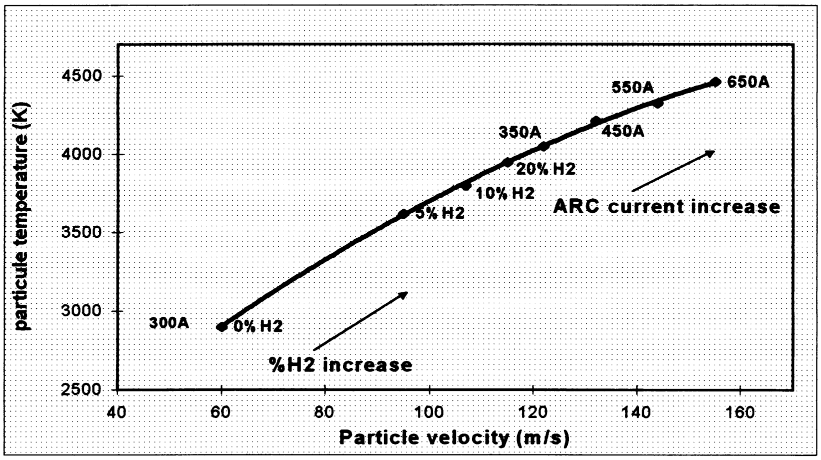

Fig. 3 Comparison of the mass lost at injection and by vaporization: effect of H_2 content. (Ar/H_2 45/15 slm, nozzle i.d. 7 mm, 380 A, 56 V, iron particles 14–55 μm).

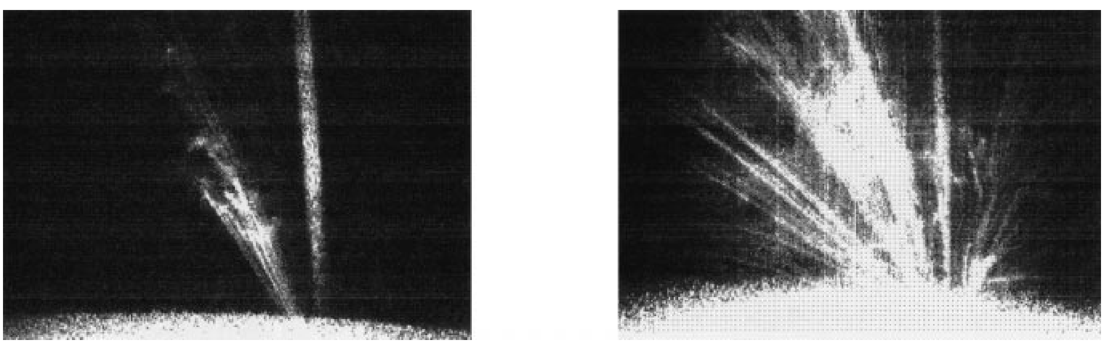

Fig. 4 Impacts of alumina droplets on alumina substrate at 1500 K.

Particles in a splashing mode can be observed. The main track of the impacting droplet is surrounded by several trajectories of ejected material depending on surface roughness. Researches are in progress to find a good mode for deposition avoiding splashing resulting in a non negligible mass loss and weak adhesion. It should be kept in mind that those results were obtained with a high substrate temperature and then a slow solidification.

Splat formation depends on the impacting droplet velocity, size, molten state, impact angle, substrate roughness and temperature, as well as its surface chemistry especially its oxidation stage [20]. For particles impacting orthogonally to the substrate with much higher temperatures than the latter ($\Delta T > 1000$ K), it has been observed in different laboratories, that the substrate temperature plays a major role in splat formation. Above a certain transition temperature, the splats solidify when flattening is almost completed and exhibit a regular disk shape on a smooth substrate ($Ra < 0.3$ μm). Below this temperature the flattening droplets begin to freeze before flattening is completed and part of the liquid splashes away resulting in extensively fingered splats. This transition temperature plays a key role on the coating adhesion/cohesion which is drastically (2–5-fold) enhanced when the preheating temperature is over it [21]. Even if the substrate temperature is over the transition temperature, when the particles impact with an angle below 60°, the splats become extensively fingered and they can be pulled off by a perthometer tip. Correlatively, the coating adhesion/cohesion becomes very poor [22,23]. At last, the shape and adhesion of the splats depend also on the oxides formed at the substrate surface [24], i.e. on the preheating time with the plasma jet. The splat formation being so critical to the coating adhesion/cohesion properties, more experimental and numerical works are required to get a better understanding of these phenomena.

To achieve good coatings, the control of the gun spray pattern is essential in order to keep the nozzle axis as normal as possible to the substrate. This is achieved by using computer controlled robots [25]. The spray pattern together with the relative velocity torch to substrate and the powder mass flow rate control the beads overlapping and the pass thickness. The temperature at which the coating and substrate are kept during spraying is closely linked to the sprayed part shape and thickness, the pass thickness and the used cooling devices (usually air jets blown at the substrate surface).

Once the spray pattern, relative velocity torch/substrate and pass thickness have been defined the only mean to control the coating surface temperature is to monitor the flow rates of the cooling jet according to the substrate and then coating surface temperature measured with a pyrometer.

Beside the adhesion/cohesion of the coating to the substrate which increases with preheating temperatures higher than the transition temperature, the control of temperature before, during and after spraying plays an important role on the generated residual stresses. The increase of the preheating and spraying temperature increases the quenching stress as well as the expansion mismatch stress. Thus a compromise has to be found and the developed recent techniques allowing to follow continuously the stress generation during preheating, spraying and cooling [26,27] can help to find it.

At last dusts and fumes have also to be very well controlled. Small particles localized in the periphery of the plasma jet in the injection zone are sucked down by the jet and penetrate progressively with low velocities in the spray jet. These particles together with those resulting from the condensation of vaporized material, disturb the homogeneity of the coating especially between successive passes and at the interface coating–substrate, and decrease its adhesion/cohesion.

POSSIBLE SOLUTIONS

To improve coating reproducibility and reliability, there are some possible solutions related to plasma guns and the implementation of on-line control of the process with adapted feedbacks.

Plasma guns:

- To reduce the cathode wear new dopants less sensitive to diffusion have to be found [29].
- To limit arc root fluctuations, new torches with high voltage have to be designed. The use of a segmented anode results in the arc attachment at the downstream segment (anode). The longer arc obtained with these anodes exhibit higher voltage(about twice that of a classical DC torch), thus reducing concurrently the voltage fluctuation percentage. This system is already used in a commercial plasma gun [30].
- To limit the air entrainment, a Laval nozzle and/or a gas shielding can be placed at the anode nozzle exit [31].

Figure 5 indicates the main instabilities and deviations of the process

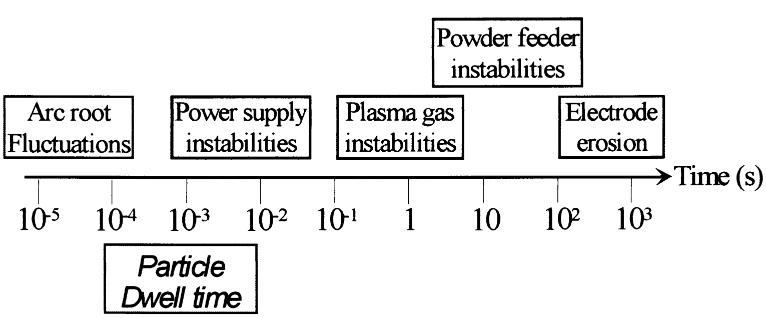

Fig. 5 Main instability frequencies in plasma spraying.

If the particle residence time is taken as a reference, the times characterizing the variations of the spray, are generally long compared to this reference time. Only the arc fluctuations have a time constant less than that of the particle dwell time. These fluctuations, as already mentioned, will affect the injection of the particles and their acceleration and heating. The others characteristic times are relatively long and it means that in a few seconds it is possible to correct the variations of the carrier gas flow rate, plasma forming gas flow rate and obviously voltage deviation due to electrode erosion.

A solution to improve reliability is the on-line control [32–34]. The key parameters, which have to be controlled on-line, are the particle spray jet position, probably the most important parameter, the particle velocity and temperature, the substrate temperature, and coating thickness. Actually if these parameters are kept in the good range the reliability and reproducibility of the process will be better. For more 20 years, various laboratories or companies have studied relationships between coating properties and input parameters. The aim of a feedback is to compensate any observed deviations of a key parameter, but it is still a challenge, because nobody according to the present knowledge, has a clear idea of the effect of the particles mean velocity or temperature at impact on the coating thermomechanical properties. Moreover control logic is complex because of interactions between input variables.

The simplest approach for the process control is to define a zone of 'correct values' and acceptable variations for the process inputs.

An example is given in Fig. 6, for which two particle parameters can be measured. According to experience it is assumed that as long as they lie in the hatched area, coating parameters are acceptable.

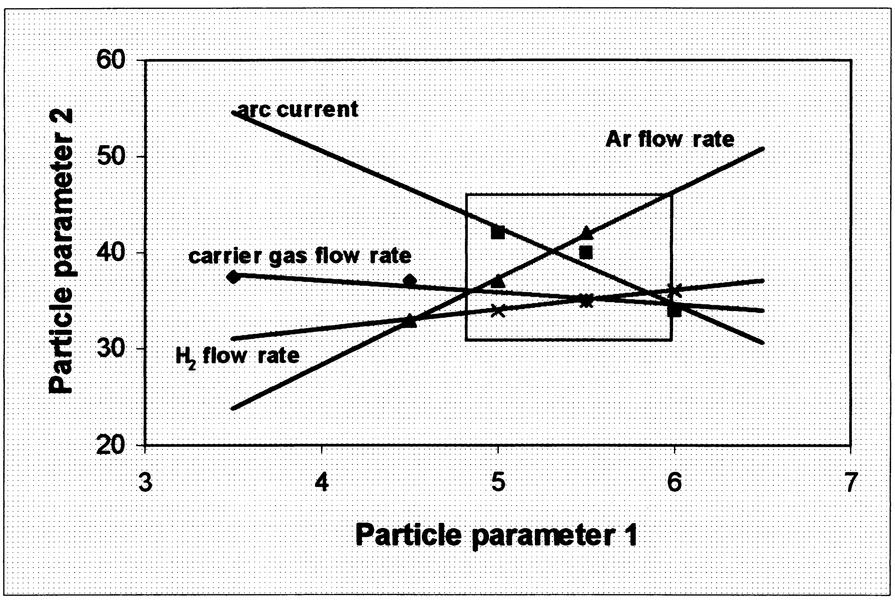

Fig. 6 First approach for a process control.

Two databases are essential to allow the feedback:

- The first comprises correlations between in-flight particle parameters and process input parameters. An example is given in Fig. 7.
- This figure shows the variations of particle velocity and temperature associated with an increase in arc current and secondary gas vol%. It seems very sensitive, but in fact the possibilities of correction are very narrow. A change in any parameter can affect the plasma stability, the electrode wear, and so on.
- The second database is related to correlations between coating properties and both in-flight particle parameters and spraying temperature. Such a database can be developed only for a given coating with specific service conditions (see Fig. 8). Concurrently it is necessary to collect the data relative to coating properties linked to service conditions (thermal barrier, wear, and/or corrosion resistance, electromagnetic shielding...), in order to create an expert system.

The corresponding strategy is presented in Fig. 9. As already pointed out previously the transfer function is linked:

- to part geometry and thickness, spray pattern and relative velocity torch/substrate, heat flux imposed to

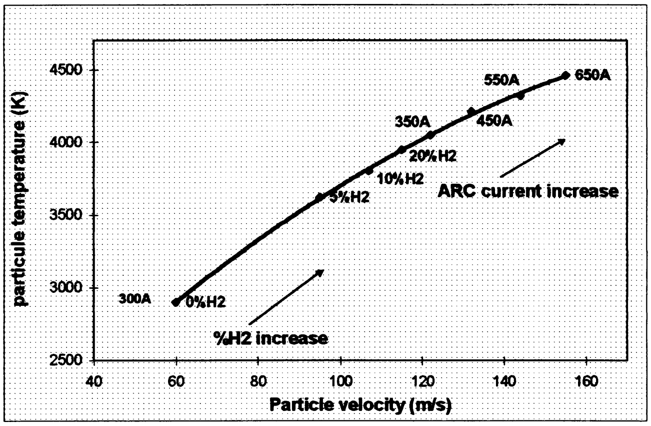

Fig. 7 Correlation between particle velocity and temperature with arc current and H_2 vol%. (TaC particles, –34 +62 μm, nozzle i.d. 7 mm, total plasma gas flow rate 90 s.l.m, stand off distance 100 mm.)

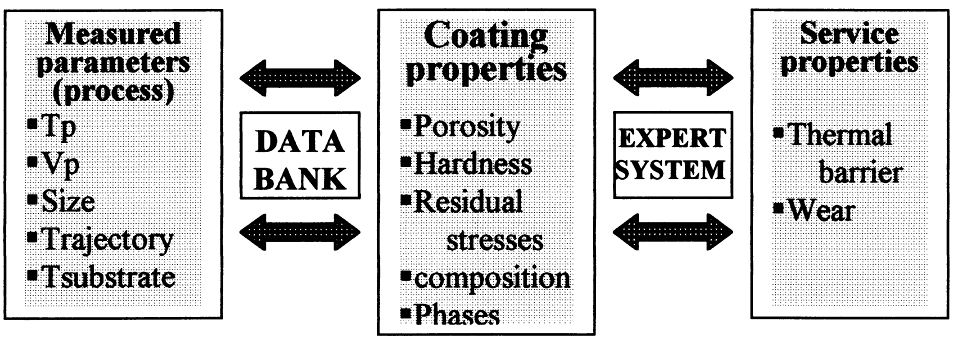

Fig. 8 Building of a database for a given coating with specific service conditions.

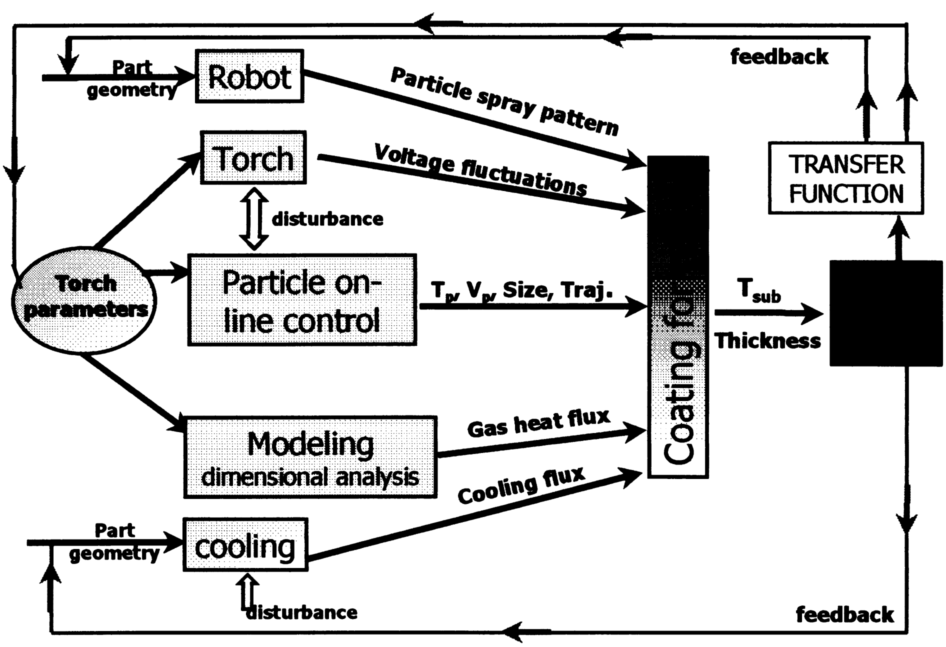

Fig. 9 Schematic of the strategy to control spray processes on-line.

the substrate by the plasma jet, cooling system parameters which control the substrate and coating mean temperature, i.e. its adhesion/cohesion and the residual stresses which, over a certain limit, are detrimental to coating mechanical properties.

- to the particles in flight mean trajectory, velocity, surface temperature…which play a key role in coating formation.

Of course beside these parameters the torch electrodes wear as well as that of the injector have to be continuously checked mainly by their consequences on the in-flight particles. For example the electrode wear, especially that of the anode, can be followed continuously with the FFT of the sound signal [35]. When this wear becomes detrimental, it can be predicted sufficiently in advance so that the electrodes can be replaced before a problem occurs during spraying. However the still pending question is which action has to be taken to compensate the voltage drop resulting from the continuous wear of the anode: increase the arc current or the hydrogen vol%.

A controller linked to the data bank collects the information and gives orders through a transfer function to correct input parameters. Various methods can be used for transfer function, but the simplest is an analytical model.

Education and training of the operators are also among the keys for the reliability and reproducibility of coatings. Among the different possibilities, to our opinion, the most important points which have to be taught are the following:

- the limitations and drawbacks of the process,
- the key-parameters of the process,
- the importance of consistent feed materials,
- the spray pattern and relative torch-substrate velocity, and the substrate and coating temperature control,
- the substrate surface preparation,
- the fumes and dust control.

Some simulation techniques could be developed for training.

Standardization is also very important for quality management and standardized methods should be used to evaluate:

- the quality of the used powder
- the preparation, spraying and machining procedures
- the evaluation of the coatings with the corresponding tests defined considering the service conditions, the labour costs, the education level of the employees and the investments costs.

CONCLUSION

The improvement of the thermal spray coating market is linked to the reproducibility and reliability of coatings.

- New sensors and controllers allow on-line control of the spray and substrate temperature. At the moment they are mainly used to monitor the process instead of controlling it. Moreover they permit the transport of the spraying parameters from one booth to another one and from one torch to another one. However their main drawback is presently a too high cost.
- For a real control, a specific data bank has to be developed to link particle parameters at impact spray pattern and substrate preparation and preheating to coating properties. However it can be done only for each coating devoted to a service condition.
- Education of the operators has to be developed and the standardization of the procedures of pretreatment, spraying, post-treatment and coating characterization has to be systematically done and used. However standardization has just started.

At last important efforts have to be made to:

- improve the torch stability
- control on-line the wear of the electrodes
- control the gas flow around the spray jet and close to the substrate
- study 'in flight' chemical reaction as well as particle vaporization and resulting vapor condensation
- a better understanding of coating formation (particle impacts and resulting splats layering)

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