Coatings

OPTIMISATION OF THE DEPOSITION PARAMETERS OF THICK ATMOSPHERIC PLASMA SPRAY COATINGS

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ABSTRACT

Plasma spray process is one of the most versatile thermal spray deposition processes. There are great number of parameters that influence the quality and characteristic of deposited coating and their values must be defined precisely for each type of coating and used equipment particularly. A review of technology developed for deposition of ferrous-based powder spray (Fe–1.3Cr–1.5Mn–1.2C–0.3Ni, wt. %) on flat Al–Si alloy substrate surfaces is presented in this paper. Possible problems that may occur before and during the deposition process were pointed out as well as adequate solutions of those problems.

Keywords: atmospheric plasma spraying, ferrous-based coatings, parameters optimisation, microstructure.

AIMS AND BACKGROUND

The surface enhancement of conventional materials that are used in tribosystems with the coating deposition is a well-established practice. Thermal spray is flexible and economically efficient procedure which enables superior tribological properties for machine elements that are designed for demanding properties. It is widely used for production of the new parts, as well as for repair purposes. Thermal spraying is, in fact, a generic group of processes in which the coating material is fed to a heating zone, where it becomes molten, and after that propelled to the substrate surface. Metallic, ceramic, cermets and some polymeric materials can be used in the form of powder, wire, or rod for this purpose! There are several different processes for thermal spray coating deposition and mostly used are flame spray, electric arc wire spray, plasma spray and high velocity oxy-fuel spray (HVOF) process².

The plasma spray process is the most widely used coating deposition method because it presents process flexibility and coating quality in combination³. This process finds wide application in aerospace, petrochemical, automotive and other industries⁴⁻⁶. Plasma spray process poses several advantages compared to the other methods of coating deposition and other surface engineering techniques. In comparison to the vapour deposition methods it requires less expensive installations⁷. Chemical treatments like electroplating of chromium and nickel coatings are becoming increasingly threatened by environmental regulation. Moreover, one should prevent microscopic particles of chromium and nickel, a health hazard, from entering the environment. Chemical treatments require complex chemistry and are relatively expensive. One of the main advantages of the plasma spray process is that the substrate is heated very little during the deposition, i.e. the substrate restrains its chemical composition, structure and mechanical properties. During the plasma spray process, introduced thermal energy melts the coating material (usually in the form of powder). Molten material is then propelled by the carrier gas onto cleaned and prepared specimen surface, where it bounds to the substrate predominantly by mechanical bonding. Detailed description of the process can be found elsewhere², while the schematic diagram of obtained coating is shown in Fig. 1.

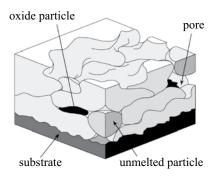


Fig. 1. Schematic diagram of a plasma sprayed-coating

Plasma spray process is one of the most versatile thermal spray deposition processes and it can be configured to spray particles over a very wide range of temperatures and velocities. The plasma temperature can reach up to $16\,000^{\circ}$ C, which enables melting and spraying of many different materials onto a wide range of substrates (metallic alloys, ceramics, polymers) and over a large range of particle size distribution (5 to 50 μ m for ceramics and 20 to 120 μ m for metallic alloys). Possible coating thickness range is also wide. Many

problems may occur during the plasma spray process like in other coating deposition methods. These problems need to be solved in order to obtain coatings with the desired characteristics. Regardless to the coating application, some characteristics are the same: coating composition and structure, porosity, presence of unmelted particles and oxide inclusions, thickness, hardness and bond strength. Coating characteristics are very dependent on substrate preparation and spray parameters. According to some researchers¹⁰, there are more than 50 macroscopic parameters that influence the quality of coating and the production of coatings is still based on trial and error approach.

The aim of this paper was to point out possible problems that may occur before and during the atmospheric plasma spraying (APS) deposition process of the ferrous-based coatings onto the Al–Si alloy substrate, as well as to give the adequate solutions of those problems.

THEORETICAL CONSIDERATIONS

Characteristics of the thermal spray coatings (metallography, hardness and tensile bond strength) were accepted as a standard one by the Subcommittee for Standardisation of Quality Control Procedures for Plasma Sprayed Coatings, which is formed in April 1997 by the European Airline Committee for Materials Technology (EACMT)¹¹. These characteristics are mutually connected and are dependent on many parameters. Generally, these parameters could be divided into 2 groups: substrate preparation parameters and spray deposition parameters.

Substrate preparation parameters affect most of all bond strength between substrate and coating. These parameters include: surface roughness and cleanliness, substrate temperature, presence of the absorb moisture and gases and presence of entrapped particles used for surface roughening. Prior to the deposition the surface of the substrate is roughened and activated so the bonding area is increased. Parameters that are important in choosing the abrasive for surface roughening are: type, size, shape, purity and hardness. Impact pressure and angle are also important. Inadequate surface cleanliness and presence of entrapped abrasive particles decrease the bond strength, so they should be avoided. Inter-layers are also used to increase the bond strength.

Substrate temperature can be important for presence of cracks, i.e. during cooling, if the expansion coefficient (and hence the contraction) of the substrate material is appreciably less than that of the coating, stresses build up and the coating may crack. That is why in some cases substrate preheating is welcomed. Slow cooling after the deposition is also good practice, regardless of the composition of substrate material. If the coating is cooled rapidly it will contract while the core is still hot (and hence expanded) and cracking may occur. Presence of the absorb moisture and gases can increase the porosity of coating, i.e. any foreign matter, on or just below the surface of substrate material, which tends to evolve gas during deposition can cause bubbles. This is frequently a problem with porous castings which are difficult to clean thoroughly, but it can also result from rolled-in contaminants in the substrate material or from contaminants in the abrasive used for roughening.

There are many spray deposition parameters, and the most commonly used are: type, flow and pressure of primary and secondary plasma gas, electric current, powder feed rate and spray distance. Speed of a spraying gun and substrate holder speed, maintained substrate temperature during the process and number of

passes (coating thickness) are also influential. All these spray parameters directly influence the bond strength, amount of unmelted particles and precipitates, porosity and type and amount of formed oxides.

Powder feed rate and spray distance are responsible for the amount of unmelted particles and precipitates. If the feed rate is too big or the spray distance is too short the powder particles do not melt completely, which results in a high amount of unmelted particles. As with all thermal spray coatings, sprayed under normal conditions, tensile stresses exist prior to deposition. These stresses increase with coating thickness. If coating to substrate bond is not adequate it can be easily destroyed by shear at the edges. One of the ways to avoid this is to blunt the edges if it is possible. Bonding strength between layers of the coatings is also important. Weaker bonding between layers is more pronounced with thick coatings and with coatings deposited with pauses. Oxidation of the coating surface is possible if there are pauses so it is recommended to continually perform the deposition.

Presence of unmelted particles and precipitates decrease the coating characteristics. Porosity shows similar effect although residual porosity of the plasma coating could also reduce the coefficient of friction through a micro-cavity lubrication system, where micro-cavity serves as a lubricant reservoir allowing improved lubrication process¹². Presence of oxides has dual effect, positive and negative, depending on the type of oxide and some other properties. In our case (ferrous-based coatings deposited by APS) the presence of FeO (wustite) and Fe_3O_4 (magnetite), as solid lubricants, improves the tribological properties of the coating. The formation of Fe_2O_3 (hematite) should be avoided, because it acts as abrasive.

EXPERIMENTAL

Materials. Substrate material was an Al–Si alloy (EN AlSi10Mg). The substrate material was fabricated using send casting, followed with solution annealing at 540°C with 35°C/h, water quenching and artificial ageing at 160±5°C for 6 h. Spray powder used in this experiment was 'Sulzer Metco 4052', which is commercial brand names of Sulzer Metco Inc. The chemical composition of powder is shown in Table 1. The powder shows fine spherical morphology with size less than 38 μm in diameter.

Table 1. Chemical composition of used powder

Element	С	Mn	Cr	Ni	Fe
(wt. %)	1.2	1.5	1.3	0.3	balance

Substrate preparation parameters and spray deposition parameters. Atmospheric Plasma Spraying, with METCO 7MB Plasma Flame Spray Gun, was utilised in the experiment. Before the spraying process, the surface of the Al–Si alloy substrate was roughened and the edges were blunted with radius ≈ 0.2 mm. A specimen radial speed was 500 mm/s. Traverse speed of a spraying gun was maintained constant at 4 mm/s. In order to get an optimum coating structure certain parameters of the substrate preparation and spray deposition were varied. All the parameters are summarised in Table 2. Varied parameters (these parameters were chosen as the most influential) were as follows:

- 3 abrasives for roughening: SiO₂ (d=220–260 μ m, t≈40 s), Al₂O₃ (d=106–125 μ m, t≈6 s) and steel balls (d=600–800 μ m, t≈50 s);
 - 4 spray distances (50, 75, 100 and 150 mm);
 - 4 powder rates (2, 2.5, 4 and 4.5 kg/h);
 - 2 thicknesses (150 and 250 μm);
 - presence of additional cooling (with and without).

Table 2. Substrate preparation and spray deposition parameters

Parameter	Value*		
Abrasive used for roughening	SiO ₂ / Al ₂ O ₃ / steel balls		
Primary plasma gas (Ar) (l/min)	100		
Secondary plasma gas (H ₂) (l/min)	5		
Electric current (A)	500		
Powder carrier gas (Ar) (1/min)	37		
Powder feed rate (kg/h)	2 / 2.5 / 4 / 4.5		
Spray distance (mm)	50 / 75 / 100 / 150		
Coating thickness (µm)	150 / 250		
Additional cooling	no / yes		

^{*} Parameters used after optimisation are bolded.

Metallographic examinations. Primary purpose of the metallographic examinations was to determine the quality of the obtained coating (bonding, presence of cracks, unmelted particles and precipitates and porosity). Metallographic examinations and characterisation of the obtained coatings were done according to the Pratt and Whitney standard¹³. The microstructure of the coatings and presence of cracks were analysed with optical microscope (OM), where the coatings were sectioned perpendicular to the coated surface. Metallographic samples were prepared in a standard way applying grinding and polishing, with no etching.

RESULTS AND DISCUSSION

After numerous test samples have been produced and analysed (mainly microstructure analysis), optimum parameters for the substrate preparation and spray

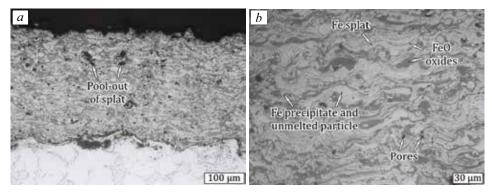


Fig. 2. Cross-section micrographs (OM): whole coating (a) and detail (b)

deposition were obtained (see Table 2). The microstructure of the optimum coating is shown in Fig. 2. Elongated splats of molten powder form a lamellar structure with oxide layers in between typical for spray coatings^{14,15}. No cracking was found in the coatings and no peeling was observed at the interface between the coating and the substrate (Fig. 2a). Big dark areas are pull-outs of splats or dirt formed

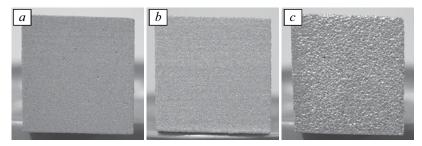


Fig. 3. Substrate surface appearance after roughing with: $SiO_2(a)$, $Al_2O_3(b)$ and steel balls (c)

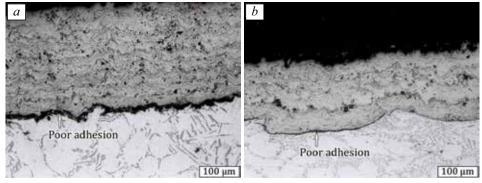


Fig. 4. Poor bonding (adhesion) of the coating with substrate in case of: (a) SiO₂ (spray distance: 75 mm; powder feed rate: 4 kg/h) and (b) steel balls (spray distance: 75 mm; powder feed rate: 4 kg/h)

during polishing. Amount of pores, oxides, unmelted particles and precipitates (Fig. 2b) were in acceptable limits for this type of coating and used equipment. Further analysis of the obtained coating, i.e. investigation of the mechanical and tribological properties of coating (which is not presented in this paper) confirmed the quality of the coating (and hence the choice of the used parameters).

First thing in the optimisation process was to ensure adequate bonding between the coating and the substrate. This bonding is mainly influenced by the

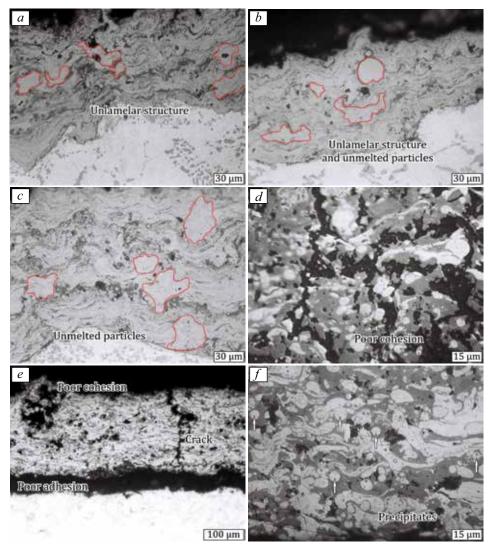


Fig. 5. Inadequate structure and poor bonding in case of unsatisfactory spraying parameters: (a) and (b) spray distance: 50 mm; powder feed rate: 2.5 kg/h, (c) spray distance: 75 mm; powder feed rate: 4 kg/h and (d) and (f) spray distance: 100 mm; powder feed rate: 4.5 kg/h

substrate preparation. Appearance of the substrate surface after activation with 3 different abrasives is shown in Fig. 3.

Substrate activation with SiO_2 and steel balls in most of cases showed inadequate bonding, i.e. adhesion of the coating to the substrate was poor (Fig. 4). That is why the optimisation process was continued with Al_2O_3 as a roughening tool.

As it is known, if the spray distance is too short and/or powder feed rate is too big then the bonding (adhesive and cohesive) and the structure of the coating is poor (Fig. 5). Poor structure is manifested with unlamellar structure, presence of unmelted particles and precipitates and high porosity.

In cases when the spray distance was too short higher amount of heat was present in the coating and as a result we have fusing of melted particles and forming of clots, i.e. we have unlamellar structure (Fig. 5a and b). In case of relatively small spraying distance and high feed rate the structure is even worse (Fig. 5c). When the spraying distance was bigger (with the same, high, feed rate) the structure was more lamellar but still inadequate. Presence of cracks and poor cohesion together with high amount of precipitates and pores were noticed (Fig. 5d, e and f).

Variation of coating thickness did not show any significant influence on the coating structure, while absence of additional cooling showed similar effect like small spray distance and high feed rate. Even with the optimum distance and feed rate there were some common mistakes like: deposition with pauses and poor cleaning of the substrate (Fig. 6a) or additional cooling with higher air pressure than it is optimum (Fig. 6b). In the 1st case we had poor cohesion between layers and in the 2nd – too many precipitates (small parts of the original powder particles) were formed.

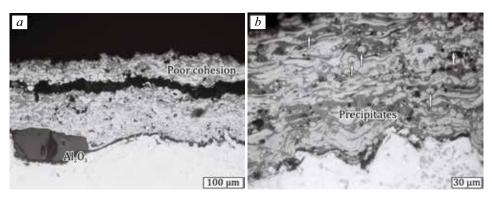


Fig. 6. Possible mistakes in coating deposition: deposition pauses was too long and the substrate was not cleaned well (a), and air pressure used for additional cooling was too high (b)

CONCLUSIONS

The aim of this paper was to point out possible problems that may occur before and during the APS deposition process of the ferrous-based coatings onto the Al–Si alloy substrate, as well as to give the adequate solutions of those problems.

After numerous test samples have been produced and analysed, an optimum parameters for the substrate preparation and spray deposition were obtained and as a result a typical spray deposition coating was produced (elongated splats of molten powder formed a lamellar structure, with oxide layers in-between).

Coating characteristic are very dependent on substrate preparation and spray deposition parameters. Many parameters influence the quality of coating and it is not easy to control all of them, so the most influential parameters should be chosen.

Unfortunately finding of the optimum spray deposition parameters (if they are not known and even in that cases, since each application requires some particularity) are still based on trial and error approach.

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