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The Influence of Dry Particle Coating Parameters on Thermal Coatings Properties

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1. Introduction

The physical properties of coatings elaborated by plasma spraying, especially the mechanical properties are strongly influenced by some fifty operating parameters of the spraying process. Several studies have been conducted to correlate these operating parameters with the coating microstructure, via the behavior of molten particles in flight to be impacted against the surface substrate, well known as splats. Then, it is expected to build coatings with tailored properties for mechanical and even thermal applications (Fauchais & Vardelle, 2000).

Simultaneously to the operating parameters of plasma spraying, characteristics of raw powder play an important role in the coating elaboration (Vaidya et al, 2001). Depending on the production process, particles feature different characteristics concerning shape, size, specific density, purity, etc. This has a significant influence on the resulting coating properties (Sampath et al, 1996). Consequently, it becomes mandatory to have an intensive knowledge about the powder characteristics in order to better control the behavior of in-flight particles and, thus obtaining coatings with the expected performance.

For the elaboration of composite coatings, it is commonly to use composite powders. However, different characteristics of powders are obtained from the variety of processes nowadays available for powder production, even for powders with the same chemical composition! (Kubel, 2000) Kubel has compared powders produced from different techniques for plasma spraying (atomization, agglomeration by spray-drying, melting and grinding, wet particle coating; sintering). A variety of powder characteristics is found for which the operating parameters for plasma spraying must be adapted to obtain deposits featuring the desired properties. From this, certain components or materials are fabricated by some of these methods or exclusively just one.

For example, when the particle shape is different, a change in powder flowability is induced. If the more spherical particles are, then powder flows much better. Consequently, the resulting properties of coatings obtained by plasma spraying of these powders are so different, even if the projection conditions, particle size and mass flow of the powders used, are constant. This is due to the difference in behavior of particle during injection and in-flight. A powder that flows with difficulty causes a blockage in the pipe injection, resulting in a decrease in the rate of deposition. Then, the overheating of substrate and detaching of coating are expected. Similarly, the injection of fine particles in a plasma jet has been a major difficulty in thermal spraying.

All these inconvenient are critical for the elaboration of composite coatings due to the great differences in physical and chemical properties between metals and ceramics. For example, when co-spraying is performed, the deposition of different phases into the coating microstructure is heterogeneous, becoming the undesired goal. For this, agglomeration of particles is the solution used to spray fine particles of metals, carbides or ceramics. Spray-drying, granulation or compression methods are used to join fine particles each other resulting in bigger agglomerates featuring higher specific area and lower density values. In certain manner, these agglomerates still retain some properties from fine particles. The result is a low rate of deposition and a porous microstructure coating because of incipient fusion of agglomerates during their passage through the plasma jet. However, particle agglomeration can easily help to prepare the metal-ceramic composite particles and get coatings composed of a metal matrix reinforced by ceramic grains. The agglomerates can be eventually, densified by sintering or calcinating and, then crushing before their thermal spraying.

To find ways of avoiding the heterogeneous deposit of different phases, coating of particles is a promising method to deposit simultaneously, metallic and ceramic phases. A variety of processes is nowadays available by including wet and dry routes. In the wet route, also known as the chemical route, a liquid phase is used to disperse organic binders in order to attach two or more different materials such as aluminum coated with nickel, or titanium carbide coated with graphite. This method seems to be used less because of the difficulties posed by the process itself, mainly by the use of organic binders, often regarded as environmental pollutants, the heterogeneity of the coating layer and the cost of production. Changes in quantity of coating material on the particles and the loss of it during spraying induce a heterogeneous distribution of phases and mechanical properties of deposits.

A new trend in the production of composite powders is actually required. This technology must be able to meet the industrial needs for manufacturing composite coatings taking into account the reproducibility of results, problems related to environmental pollution, costs and the feasibility of powders production.

In the mid-80's, Yokoyama developed the process, called mechanofusion for the production of PMMA particles coated by alumina (Yokoyama et al, 1987). Later, powders based on nickel and aluminum, were prepared by Ito (Ito, 1991). This technology allows the production of composite powders in a dry route with no need to add a binder, or sintering for attaching to the particles coating the surface of host particles. Another feature of the Mechanofusion process is the obtaining of particles with a nearly spherical shape.

Technologies for dry particle coating are relatively new and are still under research and development stages, but have a high industrial interest. In comparison with other methods for producing coated particles, the dry technique is considered "clean" since it does not use solvents or organic binder, and even water is avoided. Therefore, the cost and time of production is considerably reduced, if only by avoiding the step of drying powders.

By giving the proposal for using of two different techniques, the mechanofusion and plasma spraying, this chapter is aimed to describe how the particles characteristics play an important role in order to adapt the mechanofusion process for producing composite powders and their influence on the coatings building by plasma spraying.

2. Raw material: The particles processing

Currently, the materials used in the preparation of composite coatings can be obtained from different techniques; however, the selection of this technique will depend on the plasma spraying technique used in order to obtain the appropriate treatment of the particles in the jet and then the desired deposit. Next, it will be summarized a brief classification of composite coatings under the only criterion of the production technique of powder.

2.1 Wet coated particles

The coating of particles in a wet route is most often used for the protection of carbide powders, which decompose rapidly during spraying. This decomposition or loss of carbon causes either the oxidation of released elements or the formation of intermediate phases which degrade the deposits properties such as their oxidation resistance, hardness or wear resistance. Then, it is though that could be useful the adding a protective layer on the particles sensitive. For example tungsten carbide (WC) was coated with cobalt (Co), whose content varies from 12 to 17 wt% (Vinayo et al, 1985; Kim et al, 1997; Jacobs, 1998). Other examples, TiC can be coated with carbon or graphite (Moreau, 1990), and chromium oxide by cobalt CrO_2/Co (Lugscheider, 1992). Sol-gel is a technique for the production of composite particles at the nanoscale such as Al_2O_3/SiC system that allows obtaining deposits with some metastable phases of Al_2O_3 into the stable phase α-SiC (Jiansirisomboon, 2003).

2.2 Self-propagating High-temperature Synthesis (SHS)

The SHS process (Self-propagating High-temperature Synthesis) is part of the family of combustion reactions involving the metal reducing and oxidizing (oxygen is the oxidizing agent the most common). For the synthesis of materials by direct reaction, self-combustion is established by the exothermicity of the reaction and converts the reactants into products that are still in solid form. This does not necessarily mean the involvement of oxygen. The SHS is used for the production of composite powders containing titanium carbide (TiC), considered the best replacement for the tungsten carbide (WC) traditionally used in applications that require good wear resistance. The obtained composites coatings consist of TiC phase dispersed within a metal matrix formed by the NiCr alloy (Bartuli & Smith, 1996). In other applications, the $MoSi_2$ compound prepared by SHS is used to form a protective layer resistant to corrosion at high temperatures, such as casting nozzles in the glass industry (Bartuli et al, 1997; Gras, 2000).

2.3 Plasma spheroidized powder

Spheroidization of powders by plasma is primarily to heat and melt the particles while holding in a plasma jet. The raw materials are often milled and sintered powders with poor flowability. The spherical droplets that form are then cooled and solidified gradually. The wollastonite mineral ($CaSiO_3$) are very popular in the field of cement and ceramics, including one of its metastable phase called wollastonite TC (triclinic structure) has a great success in medical applications. Obviously, the control of chemical composition and impurities becomes mandatory. However, the irregular morphology of minerals leads to difficulties in marketing. That's why a spherical morphology of particles is desired. Work on this subject have lead to encouraging results using the plasma spraying in water, whose particle shape is spherical then this improves the flowability characteristics and the quality of the deposit (Liu & Ding. 2002; Liu & Ding. 2003). By the same principle, it is possible to improve the flow characteristics of particles initially irregulars; mixtures of milled powders of NiCoCrAlY and ZrO_2-Y_2O_3 were densified and spheroidized by plasma spraying in distilled water (Khor et al, 2000). On an industrial scale, companies like Tekna Plasma Systems Inc. produce a wide variety of powders spheroidized by plasma radiofrequency induction including powders such as YSZ/ZrO_2, Al_2TiO_6, Cr/Fe/C, SiO_2, Re/Mo, Re, WC, CaF_2, TiN (Boulos, 2011).

2.4 Atomization

As already described, spraying of particles is the most used method for the production of alloys of iron, cobalt, nickel, or aluminium (Rautioaho et al, 1996; Wang et al, 2006; Krajnikov, 2003; Kelly, 1999). But the resulting particles have no the same shape depending of the atomization media, particles may show a spherical (gas atomization) or an irregular (water atomization) shape. The deposits obtained with these powders feature an homogeneous distribution of phases. This is due to the excellent flowing characteristics of particles in the plasma and the absence of metastable phases of the compounds prepared by this technique (Sordelet, 1998; Zhao et al, 2003; Zhao & Lugscheider, 2002).

2.5 Mechanical alloying

The main objective of the plasma spraying of powders obtained by mechanical alloying is to obtain homogeneous microstructure but also very fine. Since mechanical alloying can lead to intermetallic phases that are often difficult to form even at high temperatures, the plasma spraying of powders prepared by high energy milling is then an excellent alternative for the formation of deposits of this type of composite phases. The versatility of the mechanical alloying allows processing systems such as HA hydroxyapatite reinforced with zirconia stabilized with yttria, Cu/Al_2O_3 and Ti/Al/Si_3N_4 (Fukumoto & Okane. 1992). In the case of systems with explosive materials such as aluminium powder with a very fine particle size (< 3 µm), the short-term mechanical alloying reduces the reactivity of this powder due to either the inclusion of particles of a hard phase (Al_2O_3 and/or SiC) within the Al particles or the bonding of small particles of Al_2O_3 and SiC at surface of Al (Bach et al, 2000).

2.6 Reactive plasma spraying

To reduce costs, several authors propose the use of particles capable of reacting with the environment by forming new compounds due to the reactivity of the in-flight particles and get a more homogeneous distribution of phases. Depending on the working atmosphere, the resulting species may be of oxide, nitride or carbide. Examples of this kind of coatings are those obtained from spraying of materials such as $FeTiO_3$, whose resulting deposits are composed of $Fe/TiC-Ti_3O_5$; and titanium Ti to obtain the deposition TiN, Ti_2N, TiN_{1-x}, TiC or TiC_{1-x} (Ananthapadmanabhan & Taylor, 1999; Valente & Galliano, 2000; Lugscheider et al, 1997).

2.7 Melted/sintered and milled powders

The melted/sintered and milled powders are commercially the most popular due to their relative simplicity of the production process. The main difference between the two processes is the temperature of production, which induces some differences in the properties of powders. In the case of WC/Co, sintered particles are more porous than melted ones, and the appearance of intermediate phases such as W_2C and Co_3W_3C or even only tungsten, is far more important in the melted powders than in sintered. Regarding particle morphology, melted powders are the most irregular, since the fracture is created along the crystal planes and twins while in the sintered particles, the fracture propagates between defects and grain boundaries. Obviously, resulting deposits feature different characteristics even if is the same material, such as fracture toughness and modulus are higher for deposits prepared with powders milled and blended than those sintered and crushed (Khor, 2000; De Villiers, 1998; Jacobs et al, 1998). The process of melting and milling the powder can also be used to vary the chemical composition, particle size distribution and homogeneity of the powder system (Ananthapadmanabhan, 2003).

2.8 Agglomerated powders

Apart from the more conventional technology of all, the mechanical mixing of powders [,],, the technique of preparing agglomerated powders is the most used in the field of plasma spraying of composite powders. The spray drying, commonly known as agglomeration of the powder is used to form spherical agglomerates followed by a sintering treatment in a controlled atmosphere to prevent their destruction during their penetration into the plasma jet. Different systems of powders are produced including: WC/Co, WC/CoCr, WC/NiMoCr, Ni/SiC (Wang et al, 2006; Krajnikov, 2003; Kelly, 1999; Vinayo et al, 1985; Bach, 2000; Khan & Clyne, 1996; Zimmermann et al, 2003; Wielage et al, 2001).

2.9 Other techniques

The study of development of composite coatings is not only related to the pre-processing of powders but also to different spraying protocols. For example the formation of multilayer deposits is becoming popular in applications such as deposition of thermal barrier to reduce the problem of cracking of the deposit or detaching due to thermal shock. This is also valid for the development of deposits with a combination of the properties of wear resistance and lubrication (Ramaswany et al, 1997; Gadow, & Scherer, 2001). However, if the deposit should keep just certain homogeneity of phase distribution, the co-spraying of powders allows the deposition of powders having different densities without the need for binders or pre-mix powder (Trice, 1999; Denoirjean, 2003). Another possibility for development of composite coatings is the co-precipitation of phases by melting and tempering of materials often immiscible each other (Colaizzi, 2000).

2.10 Mechanofusion

The elaboration of composite coatings using mechanofused powders was proposed in the 90's by the inventors of the system (Yokoyama et al, 1987). After that, a limited number of studies was presented in the literature. The system Ni/Al was investigated first by H. Ito et al. The system Ni/Al was investigated first by H. Ito et al. with interesting results encouraging for the industrial use of mechanofusion process as a new alternative of powder preparation for plasma spraying (Ito et al, 1991). Mechanofused powders exhibit improved flowability as compared to raw powders because of the spherical shape, which facilitates injection. Consequently, deposits are built with a homogeneous distribution of phases and the appearance of intermetallic phases formed during spraying (Ito et al, 1991; Kim, 1997; Jacobs, 1998). Several authors have evaluated different configurations of powdered systems for plasma spraying including: NiAl/TiC/ZrO_2 (Herman et al, 1992a; Herman et al, 1992b), AlCuFe and AlCuCo (Csanády, 1997), NiAl or NiCrAl-TiC-ZrO_2 (Bernard, 1994), and 316L stainless steel – α-Al_2O_3 (Ageorges & Fauchais, 2000; Cuenca-Alvarez et al, 2003a, 2003b).

2.11 An example of application

In the following sections, the influence of main parameters of mechanofusion processing, henceforth called MF, firstly on deformation of metallic particles and, secondly the particle coating will be described. The powder system is selected by considering a review of the previous bibliography oriented towards a wear resistance application.

2.11.1 Powder characteristics

Stainless steel (SS) is specified as the host particles, whereas alumina as the guest ones. The last is sustained by the increase of wear resistance by combining toughness of metals with hardness of ceramics. Physical characteristics and SEM micrographs of raw powders are given in table I. Since dry particle coating depends on the particle size distributions (PSD) of host and guest particles, PSD must be different each other at least in an order of 2 as confirmed by laser granulometry. Commercial gas atomized 316L stainless steel is provided by Sultzer Metco with two particle size distributions whereas finer α-alumina is from Baikowzki, France.

Preparation of composite particles is performed by using an in-house designed MF set-up, consisting of a cylindrical chamber rotating on the vertical axis at 1400 rpm, with a concentric joint of compression hammers and scraper blades remaining static. The gap between the inner wall of the chamber and the compression hammer is adjustable. Due to centrifugal forces and, depending on the compression gap, the powder is forced against the chamber wall and dynamically compressed through the gap. Consequently, particles bed is intensively mixed and subjected to different phenomena such as compression, attrition, frictional shearing or rolling. Then, mechanical energy input, plus the generated heat can lead to mechanical alloying, homogeneous mixing, or deformation of metallic particles.

When two different types of particles, in terms of chemical composition and particle size distribution, are MF processed; the finest particles (secondary) are attached on the coarser particle surface (host) without needing to use binders (Yokoyama et al, 1987). There are several operating parameters affecting the performance of the MF device (Cuenca-Alvarez, 2003c). However, once the characteristics of host and guest particles are specified, the key parameters are the rotation speed, processing time as a function of the powder input rate, compression gap and the mass ratio of host to guest particles. Then, a compression rate (τ) is defined by the relation between the powder bed thickness formed over the inner wall (EC) and the spacing of compression gap (EF):

$$\tau = \frac{EC - EF}{EC} \tag{1}$$

As mentioned above, this work analyzes firstly the influence of compression rate affecting the stainless steel particle shape, followed by the study of feasibility of the MF device to coat stainless steel host particles by pure alumina in function of the powder charges and the powder rate input. The corresponding variations in the operating parameters are given in table II.

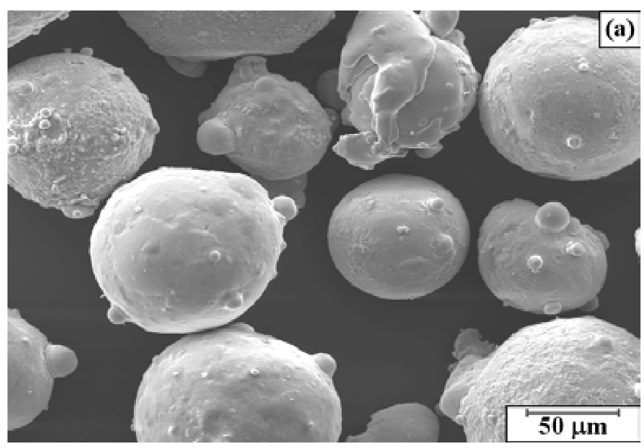
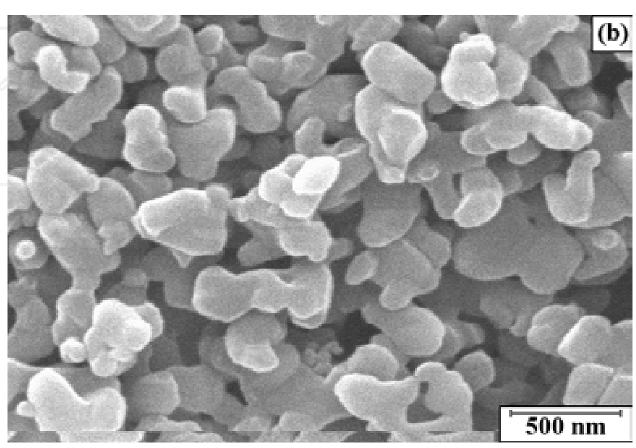
Function	Characteristics	Morphology
Host Designation: *SS*	316L Stainless Steel Mean Particle size: 142 µm Specific mass 7960 kgm-3	
Guest Designation: *AL*	Alumina (α- phase) Mean Particle size: 1.5 µm Specific mass 3900 kgm-3	

Table 1. Powder characteristics

Parameter	Value
Stainless steel charge [g]	150
Compression rate [τ]	25, 15, 5
Mass ratio of Host/Guest particle	15, 7.5, 3
Processing time [h]	1, 2, 3, 4, 5

Table 2. MF operating parameters.

2.11.2 Deformation of metallic particles

Compression rate plays an important role on the particle shape. When $\tau = 25$, metallic particles are welded to the internal chamber wall due to the overheating generated by the friction between the compression hammer and the powder bed (figure 1a). However, friction decreases rapidly at lower values of τ (15) where deformed particles are obtained as shown in figure 1b. For $\tau=5$, the compression gap is widely spaced to induce a moderate deformation of particles with a tendency to spheroidize them (figure 1c).

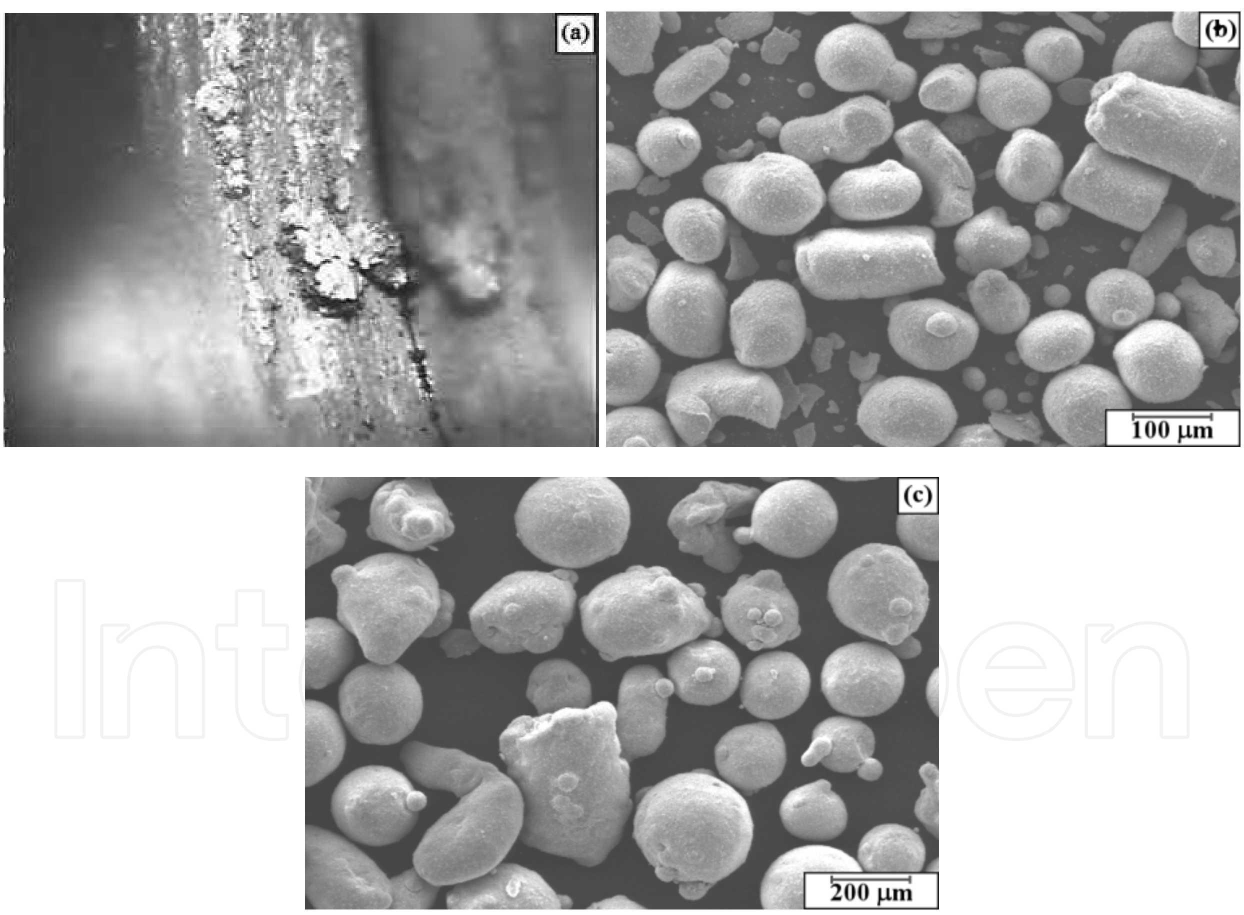


Fig. 1. Appearance of mechanofused particles at different τ: (a) 25 (b) 15 and (c) 5.

Nevertheless, fine particles (~1 µm) appear as a result of the abrasion effect taking place into the wide gap formed by the geometry of scraper blades. Thus, in order to reduce this effect, scraper blades geometry is modified to recover more efficiently the agglomerated powder from the wall surface with an incipient abrasion effect.

2.11.3 Coating of stainless steel particles by Al_2O_3

By using τ = 5, milling and overheating of particles is avoided but a rolling effect is still present, particle coating is strongly influenced by the behaviour of alumina particles into the powder bed. When alumina content is evaluated by means of processing powder at values of mass ratio of host to guest particles of 3 and 7.5, alumina particles are segregated onto the chamber wall surface as shown in Fig. 2a. This behaviour allows just the coverage of some particles featuring a heterogeneous surface coating (Fig. 2b). However, if MF process is performed with smaller amounts of fine guest particles, surface coverage is more uniform. This phenomenon applies for a mass ratio of host/guest particles of 15.0 and, is explained by a better dispersing of alumina particles within the bed of particles, avoiding their segregation.

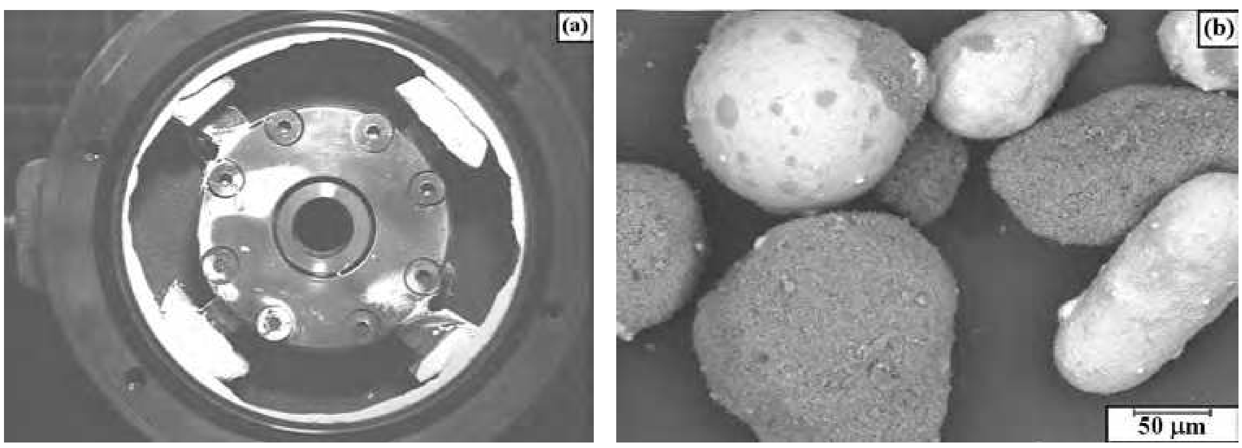


Fig. 2. (a) Agglomeration of alumina particles onto chamber components. (b) SEM micrographs of mechanofused particles at higher alumina contents.

By considering the latest, processing time is investigated as a function of the powder input rate by introducing alumina particles at 0.05 g/min in order to ensure a well dispersion of both phases into the powdered bed. Samples are taken by intervals of 1 h up to 5 h. A comparison in particle size distributions of mixtures processed at different periods (Fig. 3a), shows a slight difference in the main peak centered at 105 µm. It is likely that, even though the compression gap is widely spaced, a strong rolling effect is still induced, thus attrition of coarser metallic particles take place in the early stages of MF processing, reducing the size of metallic particles. However, a small peak is observed in the range of 0.3 to 1 µm for the samples processed up to 4 and 5 h. This phenomenon suggests that guest alumina particles, which previously have been attached to the surface of host particles, now are detached due to their successive passing throughout the compression gap. In Fig. 3b, the corresponding XRD patterns reveal an increase in size of Al_2O_3-α peak as more alumina particles are introduced. Nevertheless, a slight oxidation of stainless steel particles is detected on 47° 2θ in all cases as a result of attrition taking place in the early stages of processing described before. Then, material not oxidized is renewed at the surface of metallic particles, but oxidation does not continue because of attaching of alumina particles onto that metallic surface preventing its wearing.

Attrition and deformation effects, described above, lead the composite particles to adopt a spherical shape after 4 h of processing, achieving a shape factor of 1.25 (1.0 corresponds to the perfect sphere). Morphology and cross-sections from the resulting composite particles (fig. 4) reveal the formation of a uniform coating of alumina onto the surface of stainless steel particles, attaining up to 5.4 μm of thickness.

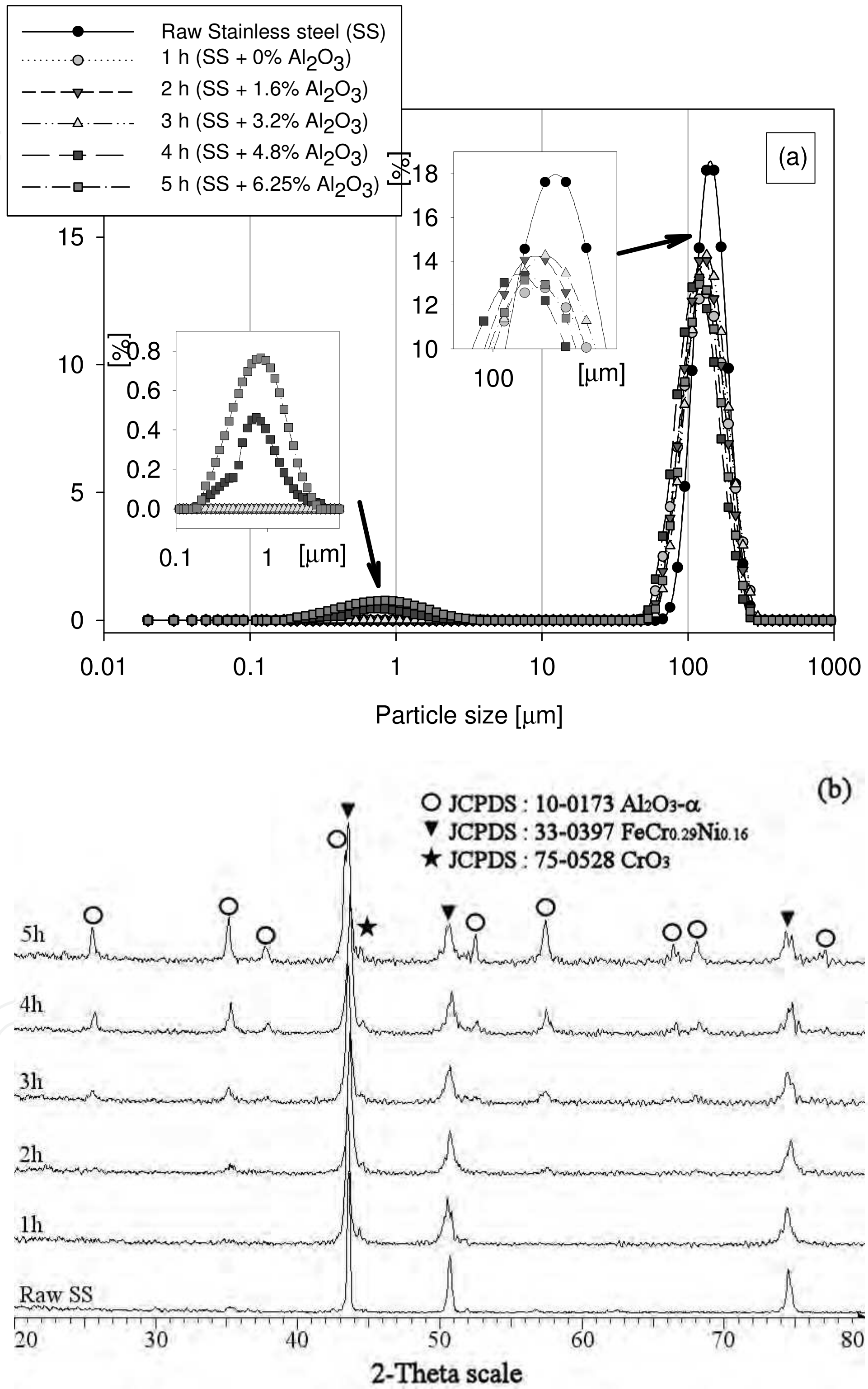


Fig. 3. Evolution of (a) particle size distribution and (b) XRD patterns during MF process of stainless steel SS plus alumina at different processing times: 1, 2, 3, 4, and 5 h.

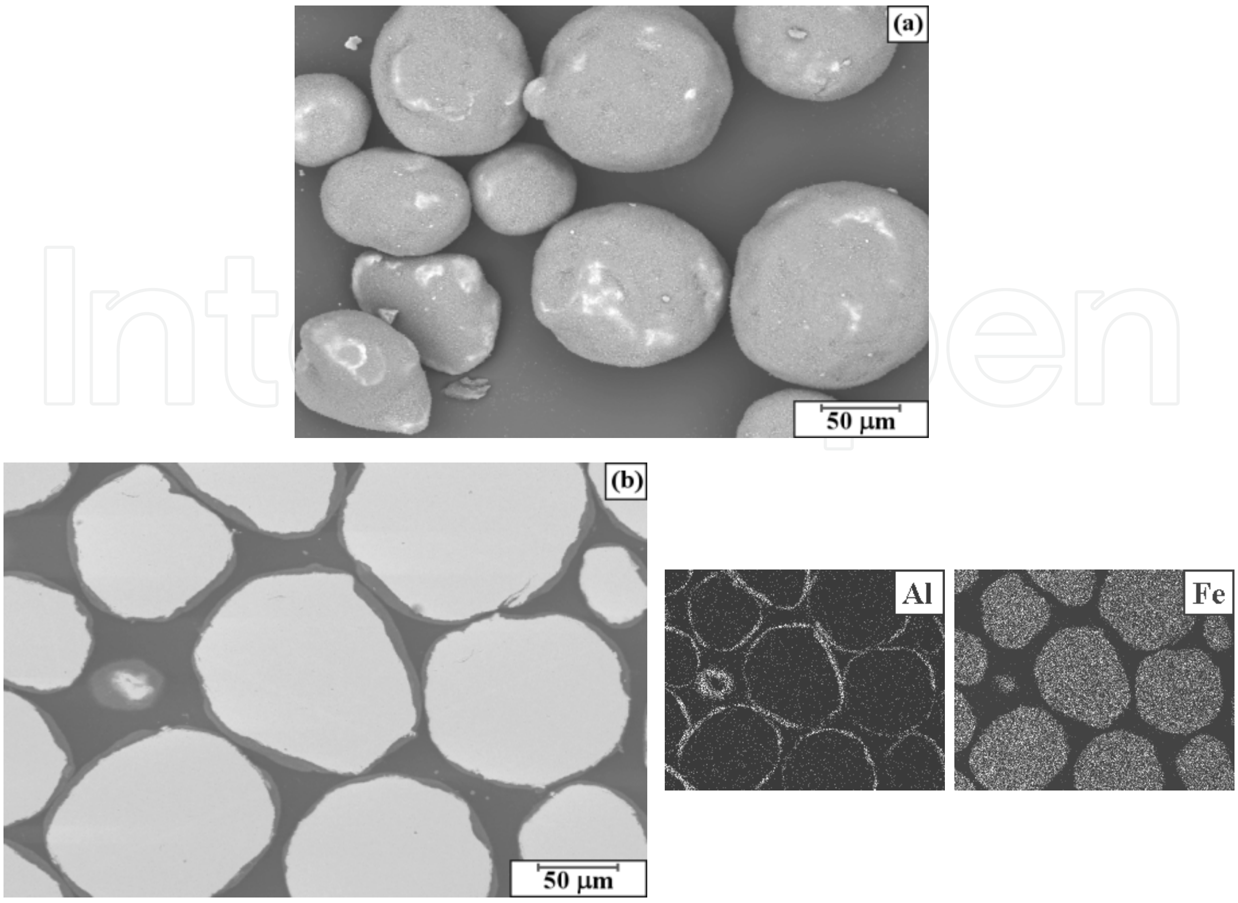


Fig. 4. Evolution of (a) PSD and (b) XRD patterns during MF process of stainless steel SS plus alumina at different processing times: 1, 2, 3, 4, and 5 h.

2.11.4 Coating of stainless steel particles by Al_2O_3 and SiC

The study for preparing a metal/oxide/carbide composite powder was performed by working with the same operating parameters indicated on table 2 but adding up to of 4 wt % alumina and 1.6 wt % silicon carbide. After 6 h of processing, the resulting particles were mesh sieved between 40 - 200 µm.

Samples of SS/AL/SiC mechanofused-composite powders consist also of a stainless steel core uniformly coated by a ceramic shell composed by a mixture of Al_2O_3/SiC. Typical morphologies and cross sections of these powders are shown in Fig. 5. All composite powders are found to be nearly spherical with a mean shape factor of 1.05 and the ceramic shell thickness attains 3.6 µm in thickness. No phase transformation or contamination was detected after the mechanofusion processing as confirmed by XRD analysis.

3. Plasma spraying: The operating parameters

Metal, ceramic or composites coatings, produced by plasma spraying are formed via the stacking of impacted particles at a very high speed (100 to 350 m / s), then flattened due to a molten or plastic state, over the surface of the substrate to be coated. The microstructure of these deposits depends on the particle behaviour in-flight into the plasma and at the impact against the substrate which was prepared previously to certain characteristics.

Simultaneously, this behavior is majorly controlled by the spraying conditions and the thermophysical properties of plasma gas. In the following, it will be presented the resulting composite coatings from the previously described powders processed by mechanofusion considering the main operating parameters of the spraying process.

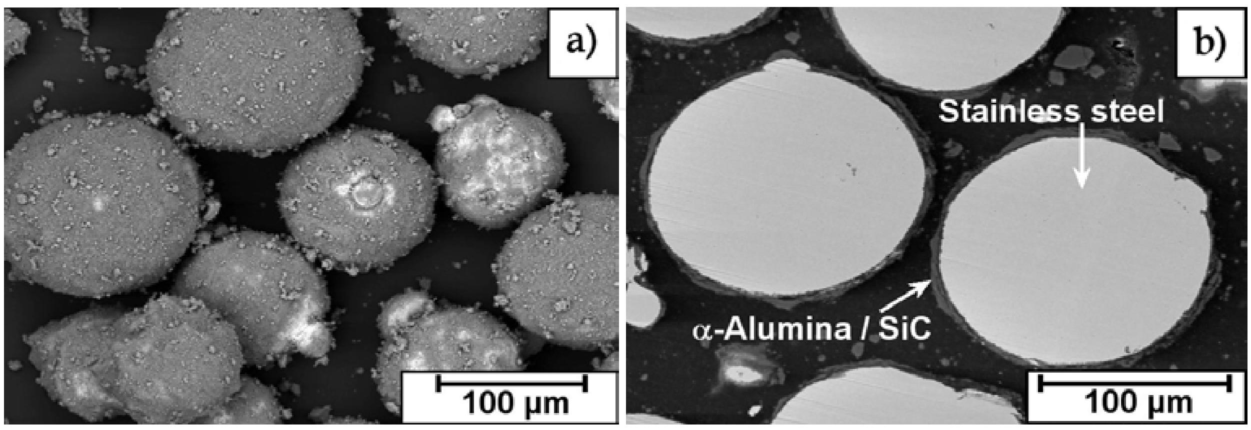


Fig. 5. SEM micrographs of (a) morphology and (b) cross section of Stainless steel/Al_2O_3/SiC mechanofused-composite powders.

3.1 Generation of plasma jet

The plasma jet is formed from a high voltage discharge (5 - 10kV) and high frequency (few MHz) between the tip of a thoriated tungsten cathode (2% Wt Thoria) and the wall of a nozzle-anode electrolytic copper (concentric to the cathode). Thus, a plasma jet flows trough out of the nozzle at high speed (between 1000 and 2500 m/s) and a temperature between 8000 and 14000 K, for an enthalpy about 100 kWh/m3. The plasma gas at the nozzle exit, have a low density (1/30th of the density of cold gas) and viscosity at 10 000 K could be ten times higher than the same mixtures at room temperature. For this case, air plasma spraying (APS) was performed with a conventional d.c. plasma torch under the parameters listed on Table 3.

3.2 The convective motion inside the particles

Inside the plasma jet, a strong movement is induced at the interface liquid-gas due to a significant difference in velocity between the fluid and the molten particles, forcing the displacement of material within of droplet. This is evidenced by the appearance of waves on the surface of the particles and oxide nodules in their core, after passing through the plasma jet (the Reynolds number is 20 to 40) (Espié, 2000). Figure 6 shows the morphology of particles collected at 100 mm downstream of the nozzle exit. Three types of behaviours, according to the state of heating of composite particles, can be observed: (Fig. 6a) those corresponding to particles just over the melting temperature where the alumina shell is broken due to the large difference of expansion coefficient between both materials (for stainless steel is 17 x 10-6K-1 and 8 x 10-6K-1 for alumina); (Fig. 6b) those more heated than in the preceding case but where the molten alumina shell was not entrained to the tail of the in flight particle, then the alumina shell is already consolidated but the host particle is burst into pieces. The third case (Fig. 6c) corresponds to the overheated particles where the light alumina shell at the surface of the molten stainless steel droplet is entrained either to the front or the back edge of the moving droplet.

Anode nozzle i.d. [mm]	7
Arc current [A]	550
Voltage [V]	57
Argon flow rate [slm]	45
Hydrogen flow rate [slm]	15
Gun thermal efficiency [%]	56
Injector external position [mm]	x = 7.5 z = 3.0*
Injector i.d. [mm]	1.8
Spray distance [mm]	100

Table 3. Plasma jet parameters

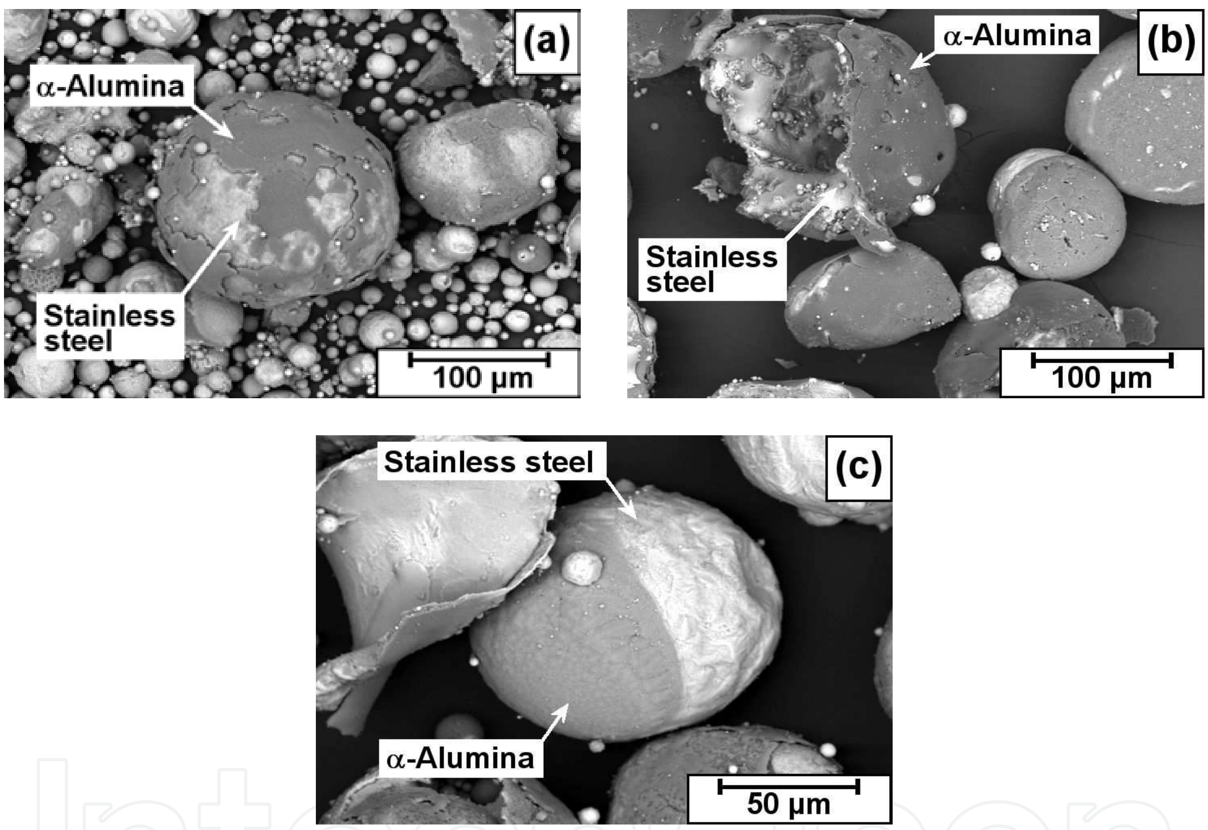


Fig. 6. Aspect of different types of particles collected in mid-flight: (a) semi-solid composite particle, (b) ceramic shell still remaining consolidated and (c) completely molten composite particle.

3.3 Chemical reactions of particles in-flight: oxidation and/or decomposition

If thermal spraying is performed at atmospheric pressure in the open air, the plasma jet is mixed with the entrained air, having different physicochemical properties, especially density is 30 or 40 times higher, inducing the formation of vortex rings. Thus, coalescence of these vortices is expected to form large amplitudes, showing difficulty to mixing with the air jet, as 'dense particles', until the plasma is correlatively cooled by heat exchange. Consequently, changes in the distributions of temperature, velocity and composition of the plasma conduce to a heterogeneous treatment of particles and, in particular it is likely to react with the entrained oxygen. The reaction rate depends mainly on the oxygen content in their neighbourhood, the exposure time of metal particles to oxygen and the particles temperature (Vardelle et al, 1995).

The oxidation reaction is developed through two mechanisms (Espié, 2000):

- By diffusion of oxygen from the surface to the core of particles (very slow, thickness about one hundred nanometers to a few microns for metals such as pure iron) that represents 1 to 2% by weight oxide;
- By convection from the particle surface towards within. In a continuous circulation, this induces the introduction of metal oxidized and dissolved oxygen inside the core of particle and the refreshing of metal to the surface. Then, the formation of oxide nodules is expected with much higher weight percentage of oxide (12 to 14 wt% for iron, for example), as compared to that obtained by diffusion.

Obviously, oxidation phenomenon inside the plasma have a significantly influence on the composition, microstructure, properties and performance of the deposits obtained. Typically, this is responsible for the appearance of defects in lamellae cohesion; chemical differences on the surface and on the coefficients of dilatation that eventually degrade the mechanical and thermal properties of deposits. The only way to prevent or slow down their occurrence is the isolating of spraying process in vacuum chambers or in controlled atmosphere, but their use remains limited to applications that justify the significantly higher installation cost (by a ratio 10 to 25).

XRD analysis (Fig. 7) reveals a slight oxidation rate of the metallic phase when spraying SS/Al_2O_3 composite powder. It is worth noting that ferrochromium oxides promote fractures and cracking when coatings are subjected to compressive stress (Volenik, 1998).

3.4 Coating building

The deposit is built by a series of successive passes that allow the deposition of particles in a melted or plastic state. The stacking of particles begins on the substrate surface and then continues on particles already deposited and, generally, solidified. Consequently, the contact conditions between lamellae/substrate and lamellae/lamellae are critical for the final properties of coatings (Bianchi, 1995; Branland, 2002). The time between two successive passes must be also considered because, for small size parts is about a few seconds while for larger parts (15 m long) this time can reach even several tens of hours.

Obviously, the final properties of coatings are directly controlled by factors concerning particles (kinetics, viscosity, chemical reactivity of droplets, temperature) and substrate (chemical asset, temperature, roughness). This is explained from a variety of studies that is found in references (Léger et al, 1996; Sampath et al, 1996; Fauchais et al, 2004; Pech, 1999).

3.4.1 Substrate temperature

From all operating parameters, the substrate temperature seems to play the most important role in the formation of lamellae. For a smooth substrate (Ra<0.05 µm), below of a substrate temperature, so-called "transition temperature, TT", the droplet breaks into interconnected pieces. The lower the temperature of the substrate, the morphology of lamellae is more irregular splash-shaped. However, over the TT temperature, the morphology of the lamellae is rather cylindrical disk-shaped with higher contact area and stronger adhesion to the substrate. It should be noted that TT depends on the sprayed and substrate materials. For example, for a zirconia or alumina on stainless steel 316L, TT is about 200 ° C.

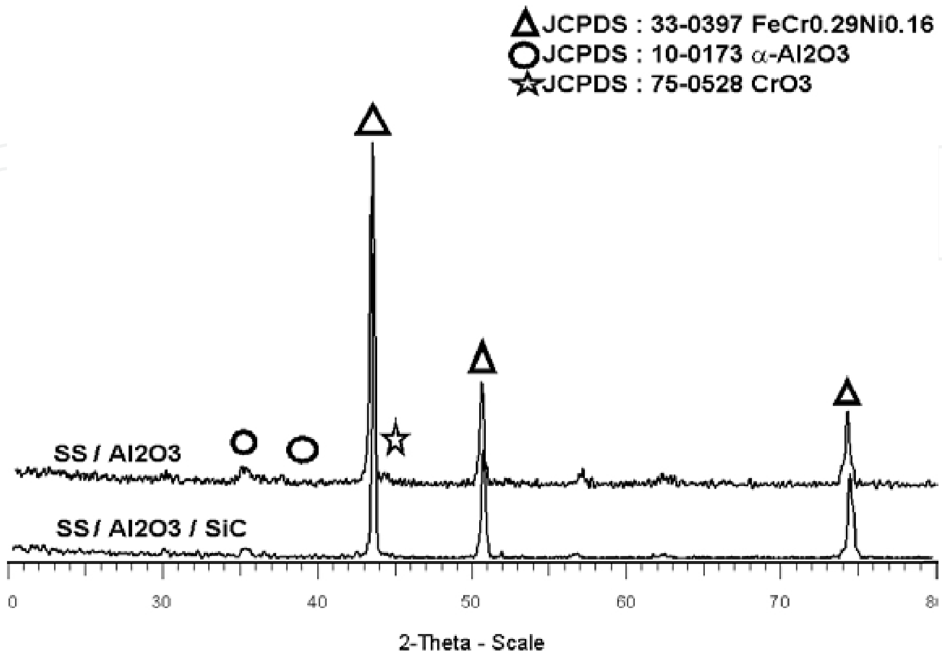


Fig. 7. XRD spectra of different plasma sprayed coatings from SS/Al_2O_3 and SS/Al_2O_3/SiC.

By considering wettability, TT also depends either on the oxidation state of the substrate and in-flight particles. If substrate is heated too long at too high temperature, an oxide layer is formed changing the characteristics of substrate surface in terms of nature, thickness, morphology and roughness of the oxide formed. Although the substrate is at a temperature greater than TT, lamellae will show a weak adhesion or even will not attach to it (Fauchais et al, 2004).

The best contact conditions observed on smooth substrates at a temperature greater than TT, also applies to rough substrates (Ra > 1 μm) and deposit adhesion is greatly increased (by a factor of 3 to 4). In addition, the morphology of lamellae also governs the size and distribution of pores, residual stresses and microstructure of the deposit.

The morphology of splats of SS/Al_2O_3 depends on substrate surface temperature. On cold substrates (TS<100°C), alumina has splashed all around the fingered stainless steel splat, as shown in Fig. 8.a, whereas a nearly disk-shaped splat, is obtained on a substrate preheated to 300°C where the aluminum is placed either over (Fig. 8b) or under (Fig. 8c) the stainless steel splat according to the host particle size. This phenomenon was explained concerning about the alumina cap position relatively to the stainless steel droplet: for the particles smaller than 100 μm the alumina cap is behind the stainless steel droplet at impact on the substrate while with particles bigger than 100 μm it is in front of stainless steel (Cuenca-Alvarez, 2003c).

For another type of splats, corresponding to particles shown in Fig 6a., alumina is scattered in small pieces over the splat surface (Fig. 8d). In-flight, the alumina pieces are either solid or close to their melting temperature i.e. very viscous. Upon flattening the stainless steel which has a high momentum pushes away the alumina pieces or maybe those beneath the flattening particle in its rim where the contact with the substrate is poor. Thus, alumina pieces are distributed evenly at the top of the splat and more regularly in its rims. It occurs whatever may be the preheating temperature of the substrate.

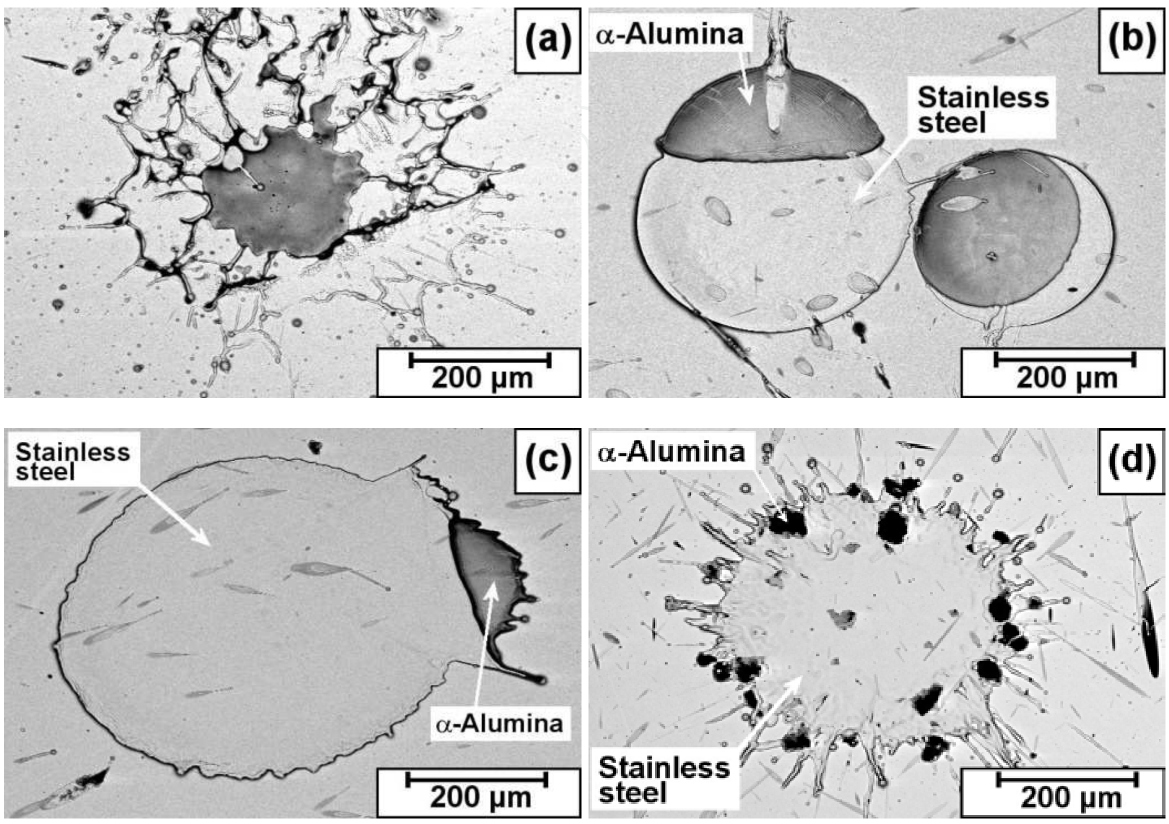


Fig. 8. Splats of stainless steel/alumina composite particles collected on (a) cold and (b,c,d) 350 °C preheated substrates.

The powdered system SS/Al_2O_3/SiC develops particular splat morphology (Fig. 9). It consist of an alumina-mesh net interconnected by fine SiC grains and distributed over the stainless steel splat as confirmed by the EDS analysis presented in white color.

3.4.2 Phases distribution and hardness properties

Typical microstructures of the resulting plasma sprayed coatings of SS/Al_2O_3 and SS/Al_2O_3/SiC mechanofused powders are shown in the Fig. 10. Both coatings exhibit a dense lamellar structure with randomly distributed hard phases within the stainless steel matrix. However the alumina distribution is coarser when spraying SS/Al_2O_3 powder due to the higher alumina content. Homogeneous distribution of ceramics is then expected either by adding a lower content of hard phase or using a smaller core particle size.

These microstructural characteristics of coatings influence their hardness properties. A comparison between the different coatings developed, illustrated in Fig. 11, shows that higher hardness is obtained with both SS/Al_2O_3 (HV5 843 MPa ± 63) while with SS/Al_2O_3/SiC is lower (HV5 756 MPa ± 38). However, the resulting hardness of composite coatings is in both cases higher than that obtained with pure stainless steel deposits (HV5

747 MPa ± 44). Two possible reasons can explain these observations: the uniformly distributed alumina within the coating and the formation of ferrochromium oxides increasing its hardness by dispersion strengthening of hard phases.

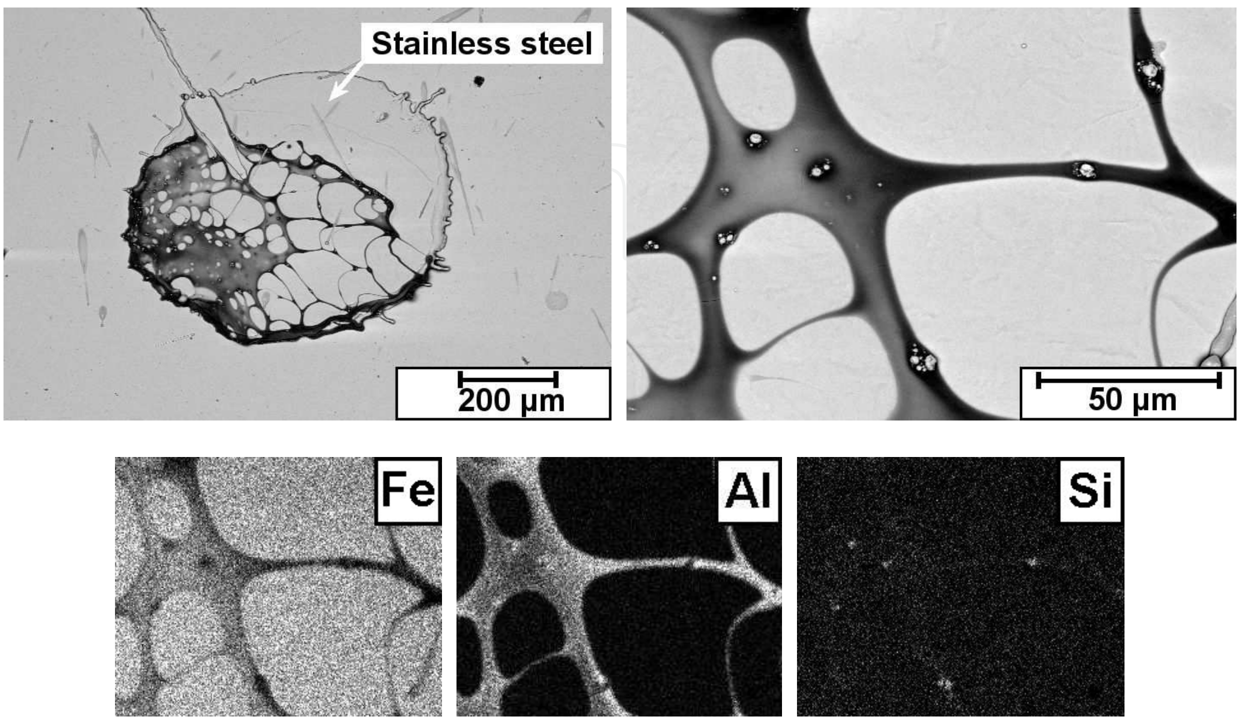


Fig. 9. Particular morphology of $SS/Al_2O_3/SiC$ splats showing the ceramic-mesh net on the stainless steel splat with their corresponding EDS analysis in white color.

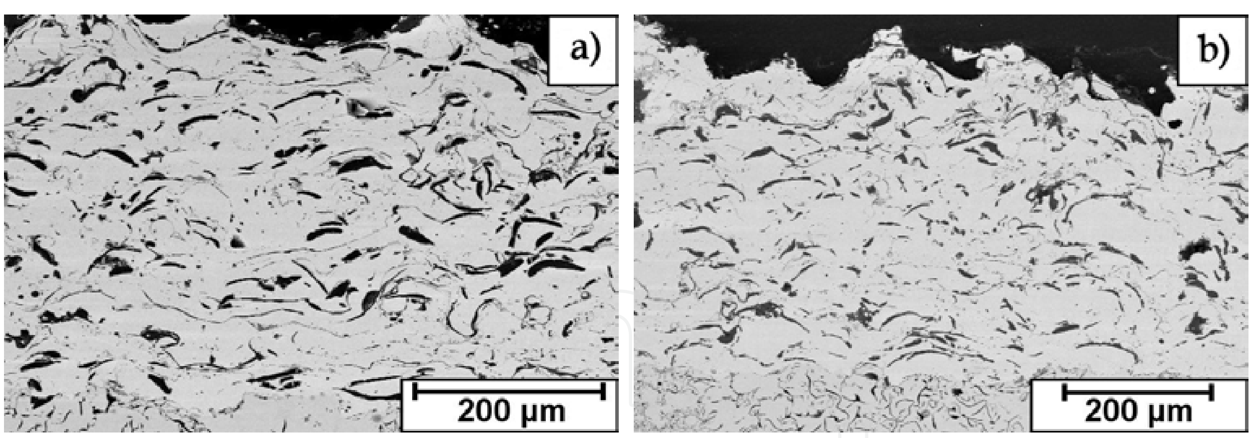


Fig. 10. Typical microstructures of plasma sprayed coatings from (a) SS/Al_2O_3, and (b) SS/Al_2O_3 SiC

By comparing with SS/Al_2O_3, hardness of $SS/Al_2O_3/SiC$ coating is lower due to the incomplete melting of particles limiting the oxide formation. It is most likely that mainly coarse host particle size and a thermal barrier effect of the alumina shell promote this state of incomplete fusion. But also, the hardness attains a value similar to that of pure stainless steel deposits. Nevertheless, no oxide formation is detected by XRD analysis with this type of composite coating. This suggests that coatings' strengthening is mainly governed by the formation of a fine ceramic-mesh net as described above in Figure 9.

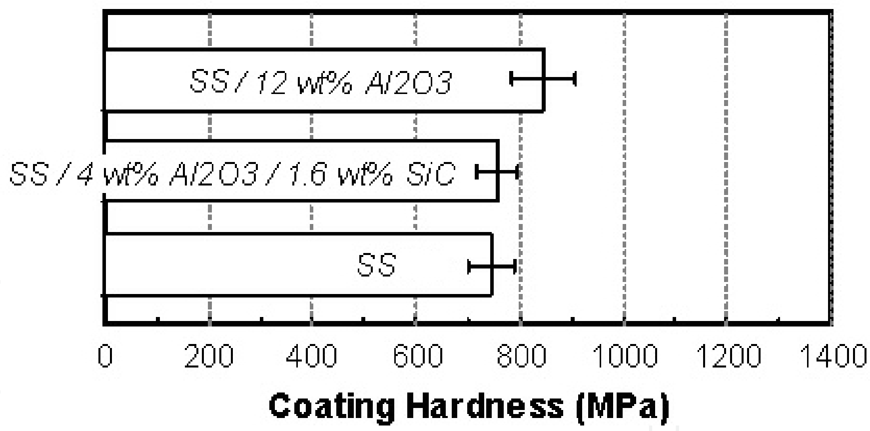


Fig. 11. Comparison of hardness between different plasma sprayed coatings developed from either pure stainless steel or composites powders.

4. Conclusions

Mechanofusion process is an effective means to prepare composite powders to be thermal sprayed and, consequently to control the plasma spray deposit microstructure. The high energy input of the mechanofusion process is directed towards the creation of particle interfaces via agglomeration of particles with a very fine size, in this case alumina (0.6 μm) and silicon carbide (3 μm), coated on stainless steel particles (–90 μm +45 μm). It is likely that agglomeration of fine alumina and silicon carbide particles on stainless steel particles is governed by the large difference in particle size distributions.

When spraying these composite powders, alumina and silicon carbide particles are found embedded and uniformly distributed in a dense steel matrix enhancing hardness properties. The final hardness is according to the kind of composite but it could be considered that the responsible for increasing the coating hardness, is mainly the uniformly distributed ceramic hard phase within the metallic matrix. Actually, the formation of ferrochromium oxides is not an option to increase coating hardness, because coarser particles (100-140 μm) are not completely melted during their passing through the plasma jet, so oxidation is still diffusion controlled

By spraying a ternary composite powder (stainless steel/alumina/silicon carbide), coating hardness is slightly higher than that of pure stainless steel. These composite coatings exhibit a particular mechanism of strengthening consisting of the formation of an alumina-mesh net interconnected by fine SiC grains and distributed over the surface of the stainless steel splat. This allows to joint directly metal splats, retaining the hard phase between lamellae.

Finally it is likely that oxidation of stainless steel particles is limited or almost stopped by their coarse particle size and a molten Al_2O_3 and SiC layer.

5. Acknowledgment

Authors would like to thank CONACYT-Mexico, SFERE-France, and National Polytechnic Institute through CIITEC, EDI & COFAA for their financial support.

R. Cuenca-Alvarez acknowledges to Teotihuacan Group for their kind help on this work.

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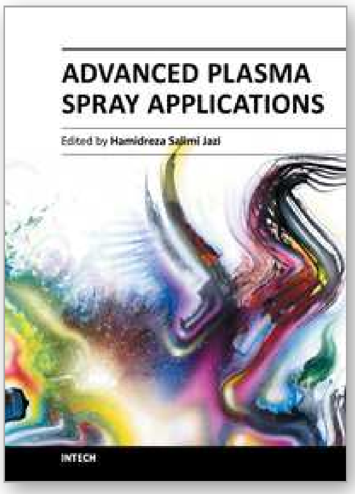

Advanced Plasma Spray Applications

Edited by Dr. Hamid Jazi

ISBN 978-953-51-0349-3
Hard cover, 250 pages
Publisher InTech
Published online 21, March, 2012
Published in print edition March, 2012

Recently, plasma spray has been received a large number of attentions for various type of applications due to the nature of the plasma plume and deposition structure. The plasma gas generated by the arc, consists of free electrons, ionized atoms, some neutral atoms, and undissociated diatomic molecules. The temperature of the core of the plasma jet may exceed up to 30,000 K. Gas velocity in the plasma spray torch can be varied from subsonic to supersonic using converging-diverging nozzles. Heat transfer in the plasma jet is primarily the result of the recombination of the ions and re-association of atoms in diatomic gases on the powder surfaces and absorption of radiation. Taking advantages of the plasma plume atmosphere, plasma spray can be used for surface modification and treatment, especially for activation of polymer surfaces. I addition, plasma spray can be used to deposit nanostructures as well as advanced coating structures for new applications in wear and corrosion resistance. Some state-of-the-art studies of advanced applications of plasma spraying such as nanostructure coatings, surface modifications, biomaterial deposition, and anti wear and corrosion coatings are presented in this book.

How to reference

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Ricardo Cuenca-Alvarez, Carmen Monterrubio-Badillo, Fernando Juarez-Lopez, Hélène Ageorges and Pierre Fauchais (2012). The Influence of Dry Particle Coating Parameters on Thermal Coatings Properties, Advanced Plasma Spray Applications, Dr. Hamid Jazi (Ed.), ISBN: 978-953-51-0349-3, InTech, Available from: http://www.intechopen.com/books/advanced-plasma-spray-applications/the-influence-of-dry-particle-coating-parameters-on-thermal-coatings-properties

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