Chapter

Atmospheric Plasma Spray Processes: From Micro to Nanostructures

Felipe Miranda, Felipe Caliari, Alexei Essiptchouk and Gilberto Pertraconi

Abstract

Atmospheric plasma spray is probably the most versatile of all thermal spraying processes, because there are few limitations either on the materials that can be sprayed or the substrate, in relation to its material, size, and shape. The material precursor of the coating could be in the form of powders, wires, melted materials, solutions, or suspensions. What distinguishes the plasma spray process from other technologies is its applicability and capacity to process a wide variety of materials, including metallic and refractory materials at atmospheric pressure. The coatings properties are improved by deposition of coatings with finer microstructure, which is are more suitable for mechanical and thermal stresses than the lamellar microstructure of conventional plasma-sprayed coatings.

Keywords: atmospheric plasma spray, solution plasma spray, thermal barrier coatings, environmental barrier coatings, nanostructured coating

1. Introduction

Atmospheric plasma spray (APS) appeared after Second World War as a surface finishing technology. It is now widely used to deposit thick coatings (from hundreds of micrometers up to a few millimeters) in a substrate to protect in aggressive environments or to improve its function. APS is commonly used in many industrial sectors, including aeronautics, energy, automotive, mining, biomedical, and electronics [1]. The synthesis of coatings by APS technique occurs by stacking the lamellae resulting from the impact, flattening, and solidification by the colliding molten particles [2]. The material precursor of the coating may be in the form of powders, wires, melt materials, solutions, or suspensions [3]. APS can be applied to a wide variety of materials, including metallic and refractory materials. In this technique, a carrier gas conducts the material particles by injecting them at high velocity through the plasma, where they are molten or partially molten, taking the form of droplets that settle and solidify on the surface being coated. The material to be deposited is carried in the form of a solution or powder to a torch with sufficient enthalpy to generate a plasma jet to melt the particles [4, 5]. The parameters of the plasma spraying process, as well as the characteristics of the precursor (solids or liquids) used for coating, influence the properties of the deposited materials [6, 7]. Characteristics of the coatings such as porosity, atomic structure, roughness, cohesion, and adhesion are fundamentally related to the interaction of the precursor with the plasma jet [8, 9].

The main driving force for the manufacture of thick coatings by APS is their high deposition rate; a few kilograms per hour of raw material can be processed with torches, with a power level of a few tens of kilowatts at a relatively low operating cost. Plasma spray is probably the most versatile of all thermal spray processes because there are few limitations of materials that can be sprayed or on the material, size, and shape of the substrate [4]. The coatings are characterized by a highly anisotropic lamellar structure. In addition, stacking of the particles generates specific interlamellar characteristics throughout the structure, especially voids, which may connect or not through the particles encountering the coating thereafter.

Conventional plasma spray processes (CPS) use powders with a particle size ranging from 10 to 100 μm. Typically, these result in coatings formed by lamellae of micrometer thickness and diameter of a few tens to hundreds of micrometers. The interest in developing and studying plasma spray coatings that have nanometric and non-micrometric characteristics has been the focus of the last 30 years. This interest stems from improved nanometric coating properties compared to micron size [10]. Reducing the structure to the nanoscale improves hardness, elasticity modulus, and thermal conductivity of the coating as well as reduces defects (voids). One of the main drawbacks in the processing of nanometric particles by APS is the difficulty in injecting them into the high enthalpy jet core. In this case, it is necessary to adjust the injection angle and the transport gas flow of the material in such a way that it is not so intense that it crosses the plasma jet or so smooth that it cannot reach the center of the jet [11]. For this reason, new plasma torches have been developed using axial injection, which is a method by which the material is injected directly into the plasma torch, ensuring that all material is processed. This feature assists in the processing of materials with nanometric magnitude, which can mainly be dispersed in liquid medium to facilitate loading and processing [12].

2. Why apply coatings?

Coatings are applied to substrates (metallic, ceramic, polymeric, or composite) to incorporate to their surface one or more characteristics or qualities that they do not originally possess, to maximize the useful life of a material/equipment, by increasing its resistance to corrosion, wear, oxidation, thermal protection, or gaining efficiency, depending on its application [13].

The improved performance of a given component means, in addition to a providing greater longevity, reduces costs related to the project and subsequent maintenance. In this context, the choice of material is as important as the design of the final product. New materials have been developed for the most diverse applications, which are subjected to extreme conditions, such as in high temperatures, abrasive wear, oxidizing and corrosive atmospheres, or even in a combination of these conditions. However, it is impossible for a single material to meet all these requirements, even for special alloys. This fact contributes to increased demand for the applied coatings, which can be deposited in substrates of most materials and with properties adjusted to specific necessities [14].

Coatings can be divided into thin and thick films. The thin films have thicknesses up to 20 μm and are typically synthesized by chemical vapor deposition (CVD) or physical vapor deposition (PVD). However, most thin film deposition processes require reactors that work at low pressures, increasing process costs and limiting the dimensions and geometry of the substrate to be coated. Another category of coatings are thick films, which have thicknesses greater than 20 μm, or even a few millimeters. These are applied when the performance of the protection depends directly of the thickness of the coating, for example, coatings applied in aerospace devices, which are subjected to severe erosion, corrosion, and oxidation environments. The deposition methods of thick films include chemical/electrochemical plating, brazing, solder overlays, and plasma spray that are performed at atmospheric pressure [15].

The evolution of these coatings is often the limiting factor in the development of a technology. For example, the input temperature of the first gas turbine (1933) was 400°C. In 2011, the Japanese company Mitsubishi Hitachi Power Systems developed an advanced gas turbine with input temperature of 1600°C, and efforts are under way to raise this temperature to 1700°C [16]. Initially this evolution occurred through the development of super leagues and the consolidation of these by directional solidification and monocrystals. The construction projects also promoted advances, mainly in the cooling through internal channels of the parts in the hot section of the turbine, where cold air is injected. However, the greatest advance in the temperature operation of these thermal machines occurred with the application of thermal barrier coatings (TBC), which are applied with APS process [17]. The TBCs (**Figure 1**) are composed of three layers: the first layer is a metallic layer (bond coat), which provides resistance to oxidation and corrosion, and has an intermediate coefficient of thermal expansion between the substrate and the ceramic layer. The second is a layer of oxide (thermal grown oxide (TGO)) that appears, along the thermal cycles, in the interface between the metallic layer and ceramic, due to the oxidation of the metallic layer. The third layer is a ceramic (top coating), which is responsible for the durability of TBC as a whole, as it promotes thermal insulation and protects the metallic layer and substrate from exposure to hot and oxidizing gases from combustion.

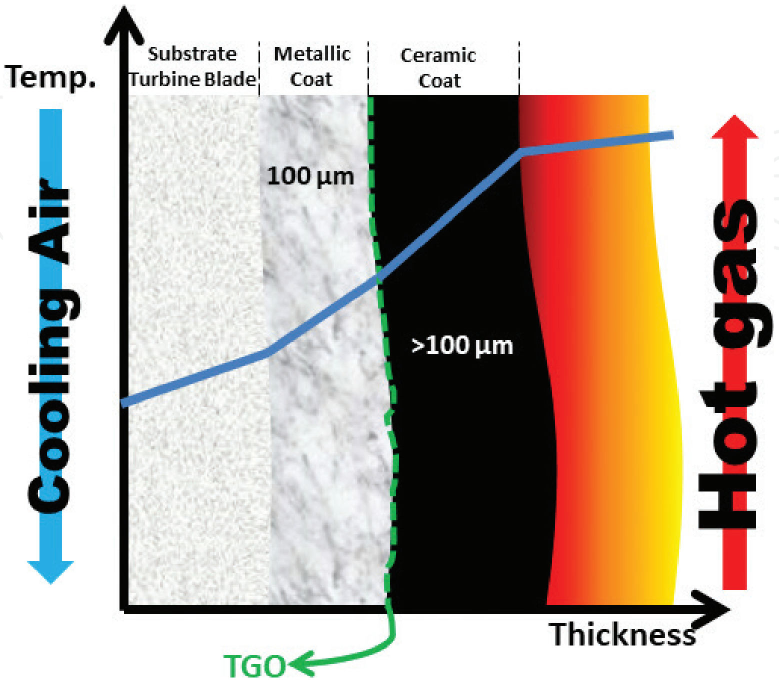


Figure 1.
Thermal barrier coatings applied on turbine blade.

3. Atmospheric plasma spray process

Atmospheric plasma spray belongs to a family of processes called thermal spray. Thus, the thermal spray process encompasses a set of processes where sprayed materials (powder on the order of μm or solutions in the form of suspensions, solutions, sol-gel or colloids) are heated (to be melted or semi-melted or partially evaporated) and accelerated against a substrate to form a coating, which may be lamellar or columnar. The thermal source may be by combustion of hydrocarbons or electric arc. The plasma spray process consists of forming the plasma jet, which interact the particles with the plasma. The plasma jet provides thermal and kinetic energy to the material to be deposited by directing it to a substrate to produce coating [18].

In the deposition process of plasma spray, the substrate is usually prepared to receive the coating by performing cleaning procedures (removing oils and greases), inducing surface roughness, preheating, and controlling movement. The adhesion of the particles to the substrate and between the lamellae strongly depends on the preparation of the substrate, preheating temperature, and morphology and composition of the material. Preheating, generally performed with the plasma jet itself, is a key point and has to be controlled according to the size and thickness of the part to be coated. Substrate and coating temperatures, either for preheating or during deposition, are linked to the residual voltage distribution, which is a controlled parameter [19].

Another important step in the deposition process is the material injection, which is mainly influenced by the injection method, feed system type, and the characteristics of the material. The injection method may be radial or axial (**Figure 2**).

In the radial injection method, the parameters to be optimized primarily include the flow of the loading gas and the position and geometry of the injector. The point is to ensure that the momentum density of the particles (product of the specific mass by velocity) is equal to the momentum of the plasma jet at the point of injection. The radial injection method has restrictions and some negative aspects, such as the heterogeneity of the heating and the acceleration of the particles, according to their granulometric distribution; and the difficulty of processing precursors with high particle velocity, because of the low plasma-particle heat transfer efficiency. The APS process should attempt to maximize the residence time of the particles in the plasma jet. To overcome the residence time problem, small amounts of hydrogen are added to the working gas to increase the enthalpy of the plasma jet; however, this also increases the electric arc oscillation [20].

Another method is axial injection, which is used in flame spray, high velocity oxygen fuel (HVOF), induction (RF) plasma torches, and some plasma torches from a direct current source (DC). In axial injection the distribution of particles are keeping more concentrated, and the interaction time between plasma-particle is

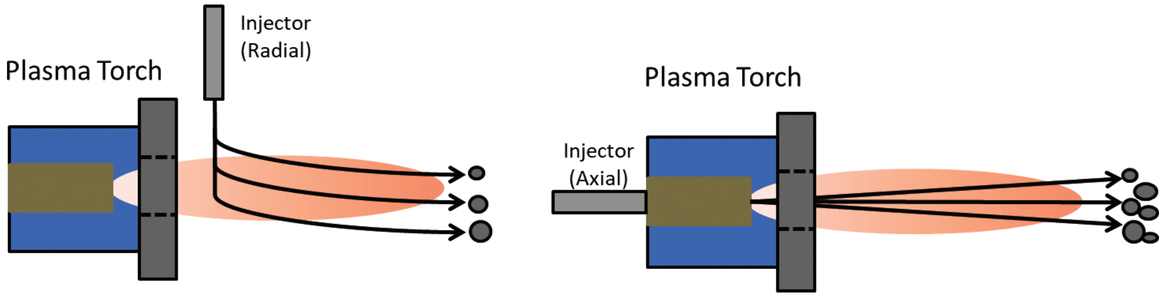


Figure 2.
Radial and axial material injection.

greater, which ensures a better processing of the material. With higher heat transfer efficiency between plasma/particle, there is a lower incidence of unmelted particles that may weaken the coating. The problems are the clogging of the nozzle and the operational stability of the plasma torch. Thus, there is a need to develop new plasma torches that operate better with this injection method.

Caliari et al. [21] presented a plasma torch with axial material injection, which can produce high velocity plasma jet (>1200 m/s). The materials are injected into the back of the plasma torch (**Figure 3**), ensuring that all material passes through hot area near of the electric arc, which is the hottest part of the torch, thereby obtaining a lower rate of unmelted particles. The process called “High Velocity Plasma Spray” has a stable torch that operates with low currents between 50~150 A and voltage of 240~360 V, unlike conventional torches, which operate at low voltages and up to five times higher current values. This differential guarantees lower erosion of the electrodes, and its geometry provides better use of the energy with close to 80% efficiency of electric to thermal energy conversion. This high efficiency enables the processing of metallic and ceramic materials, both solids or liquids precursors (suspensions or solutions) [12].

3.1 Plasma spray system

The principal components of the plasma spray systems are (see **Figure 4**): plasma torch, process control, power supply, gas supply, material feeder (powder or liquid), and dynamic sample holder. The main element of a plasma spray system is the plasma torch, which is responsible for converting electric energy into thermal energy, necessary for material processing. A process control console

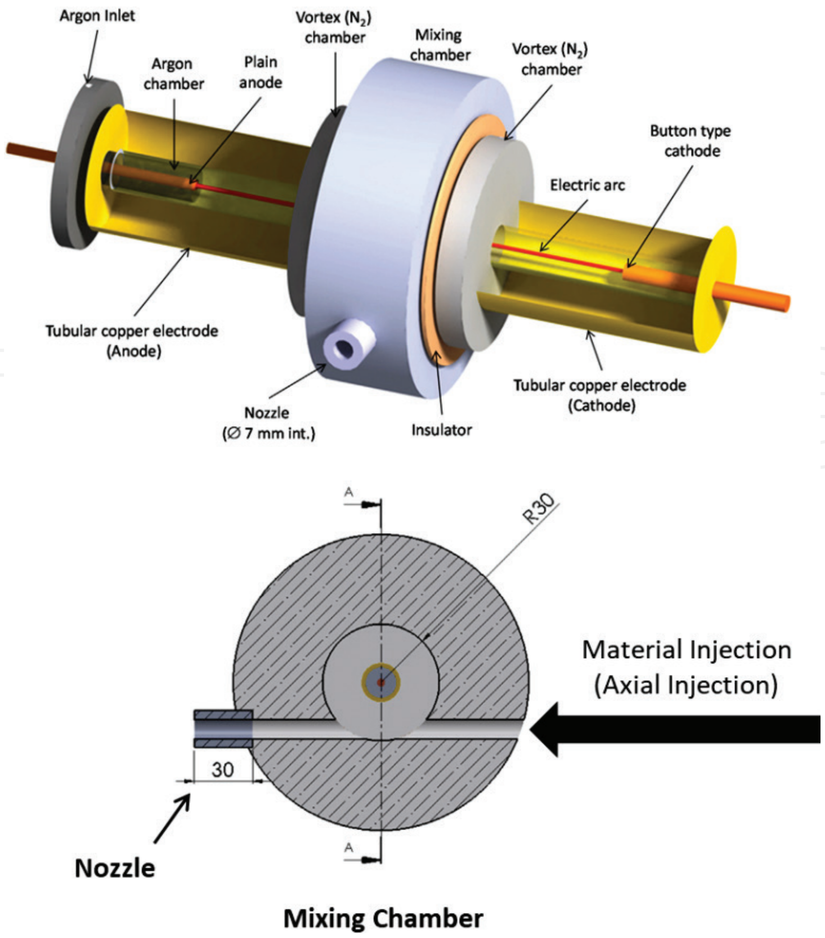


Figure 3.
High velocity plasma spray with axial injection presented by Caliari et al. [21].

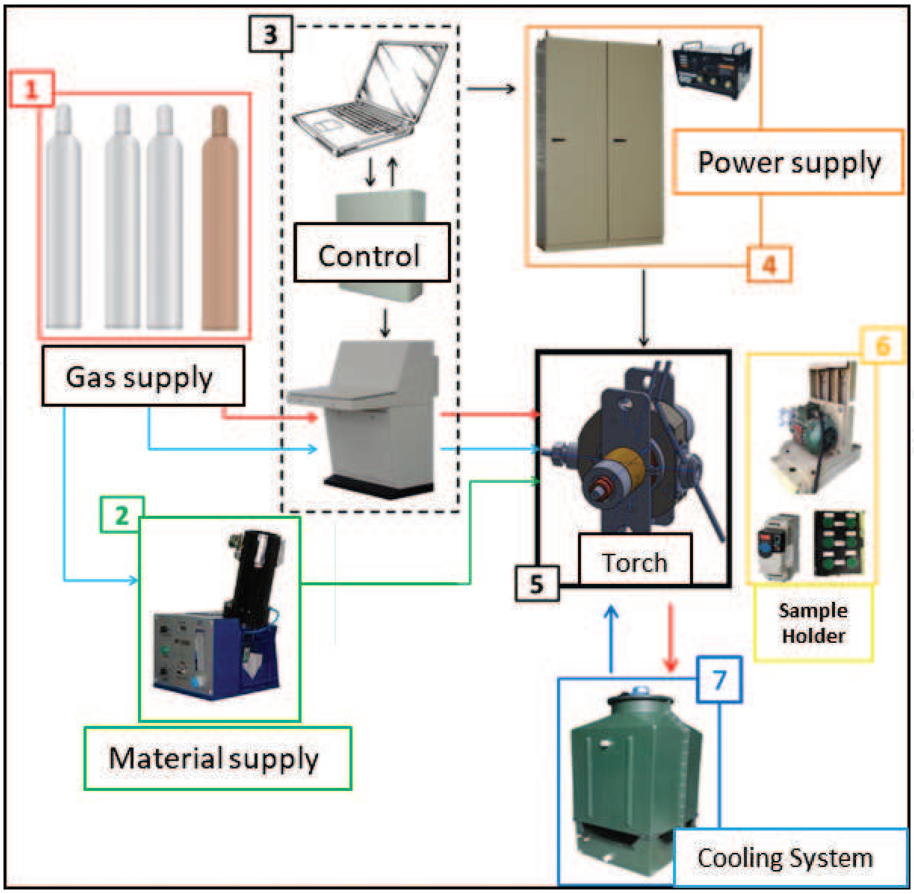


Figure 4.
Plasma spray system: (1) gas supply, (2) material supply, (3) control panel, (4) DC power supply, (5) plasma torch, (6) sample holder, and (7) cooling system.

allows adjustment of the operating parameters, i.e., the control of arc current, arc ignition, plasma gas flow rates, material and carrier gas flow rates. The additional systems necessary for operation are the plasma gas supply system, the power supply system (including the high-frequency starter unit), the high-pressure cooling water system, and the material (powder or solutions) feed system. The systems also have mechanical equipment (sample holder) to control relative motion between the plasma torch and the substrate.

3.2 Plasma torch: general aspect

Plasma torches are devices used to stabilize an electric discharge with gas flow and to convert electric energy into thermal energy. In a thermal plasma torch, operating from an electric discharge, the high enthalpy plasma results from the interaction of the gas with the electric arc. The study of electric discharges in gases and plasma jet formation involves the phenomena of gas dynamics, mass and heat transfer, and electrophysical and aero-thermodynamic processes [22].

Plasma torches can be classified according to the source of electrical energy (electric arcs generated from a direct current, DC, or alternating current, AC source) or by the type of discharge used (transferred or non-transferred arc). Transferred arc torches have one of the electrodes external to the torch body, through which the arc extends from the inner electrode. Due to the electric current transport in the generated plasma jet, this configuration forms higher enthalpy plasma jets than non-transferred arc torches. For non-transferred arc torches, both electrodes are positioned inside the torch. Thus, the electric arc remains confined in the discharge channel and the generated plasma jet does not carry electric current [23].

In the design of a thermal plasma torch, one should consider the type of electric power source, the enthalpy and temperature of the plasma jet suitable for the application, the choice of appropriate materials, the implementation of arc stabilization, and control system for the length of the electric arc (if any). In the case of non-transferred arc torches, the stabilization of the electric arc can be done with a gas vortex (that forms the plasma), discharge chamber wall, magnetic field and its combinations. In most cases the arc self-fixation method, based on shunting effect, is used. In addition, to fix the arc length, a magnetic field generated by one or more solenoids may be used. In this case, the radial part of the electric arc moves axially to the place where the axial component of the magnetic field is greater. Moreover, the interaction between the magnetic field and the electric arc current produces the driving force that displaces the arc tangentially, which avoids the positioning of the arc spot at a fixed point, thus reducing erosion of the electrode [24].

The materials applied in plasma torches, especially those exposed to the electric arc, are submitted to a high thermal load in the place of fixation of the electric arc, that destroyed the electrode. Properties such as specific heat, melt temperature, coefficient of thermal expansion, thermal conductivity, work function, and electrical resistivity should be considered in the choice of these materials.

Figure 5 presents a generic scheme of a non-transferred arc plasma torch with the electrodes (cathode, 1, and anode, 2) arranged concentrically and between the electric arc [25]. To stabilize the arc, the gas vortex formed by the vortex camera (4) installed between the insulated electrodes (3). To fix the arc in the anode surface, a magnetic field produced by the solenoids (7) is applied. The electrodes are subjected to high heat flows, and to maintain their functionality, they are cooled by a continuous flow of water through the cooling jacket (6).

Although there is a great diversity of plasma torch designs, their principle of operation is based on the generation of plasma flow due to forced convective interaction of a gas with the electric arc, established between two electrodes [23]. The application of a high frequency and high voltage at electrodes allows transform the electrical insulator gas into conductor and form an electric conductor channel. The high temperature in the channel, caused by Joule effect reduces the electrical resistance of the gas, due to an increase in the number of charged particles, thus allowing the passage of high current by the gas, establishing the arc. The electrons, accelerated by the electric field in the region between the electrodes, transfer their kinetic energy to the heavy particles through collisions, raising the temperature of the gas, dissociating its molecules, and exciting and ionizing the atoms, which are factors that contribute to the increased degree of plasma ionization. The electric field generated near the cathode accelerates the

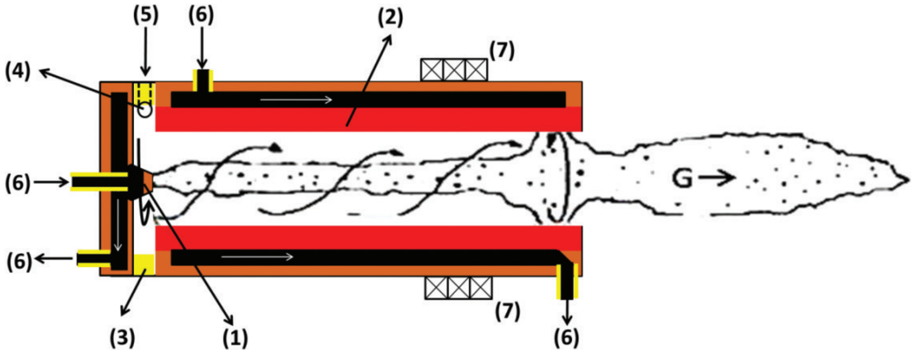


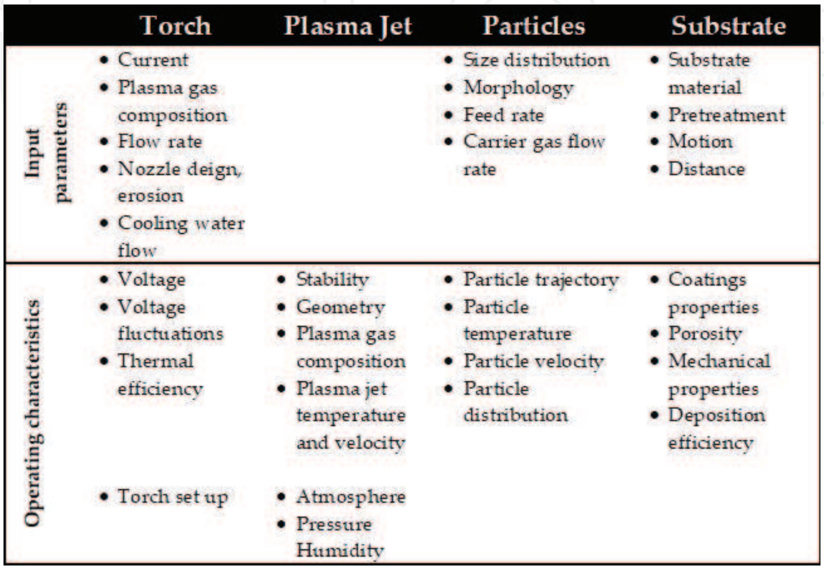
Figure 5.
Diagram of a linear thermal plasma torch, (1) hot cathode, (2) anode, (3) electrical insulation, (4) vortex chamber, (5) gas inlet, (6) cooling water inlet and outlet, and (7) solenoid.

positively charged heavy particles, which collide against the surface of the cathode. As the mobility of heavy particles is much smaller than that of the electrons, an excess of positive volume of charged particles is formed in the region near the cathode. This phenomenon increases the electric field in the vicinity of the electrode, which in turn, facilitates the emission of the electrons of the electrode (due to the tunneling and field effect) by increasing the density of electrons in the plasma [23]. The constriction of the arc in the cathode (another important phenomenon) increases the current density and, respectively, the thermal flow to the surface of the cathode, increasing its temperature and the emission of the electrons by thermionic effect.

4. Main parameters of the atmospheric plasma spray process

The input parameters and the operational characteristics of the plasma spray process are described in **Table 1** [15]. The input parameters are controlled during the experiment and, therefore, are independent variables. However, the operational characteristics often depend on the combination of the input parameters, thus, they are dependent variables.

To obtain operational control of the plasma torch, it is necessary to know the operating range of the input parameters in order to stabilize the electric arc. Hence, one must know the current-voltage characteristics (SVS) (voltage—versus current for different flow rates) of the electric arc, (CVC), which usually influenced by the characteristic curve of the electric power source. According to Heimann [26], the most common gases used in plasma torches are nitrogen, argon, helium, and hydrogen. Gas flow and its chemical composition, current and outlet electrode (nozzle) design directly influence the energetic and kinetic characteristics of the plasma jet and its stability. The arc voltage, in turn, depends on the gas flow rate, plasma torch geometry and mode of arc stabilization. Thermal efficiency represents the ability to convert electrical energy into thermal one (enthalpy of the plasma jet). Part of the thermal energy is dissipated in the electrodes with cooled walls. In conventional plasma torches, the thermal efficiency is approximately 50% [15].



	Torch	Plasma Jet	Particles	Substrate
Input parameters	• Current • Plasma gas composition • Flow rate • Nozzle deign, erosion • Cooling water flow		• Size distribution • Morphology • Feed rate • Carrier gas flow rate	• Substrate material • Pretreatment • Motion • Distance
Operating characteristics	• Voltage • Voltage fluctuations • Thermal efficiency	• Stability • Geometry • Plasma gas composition • Plasma jet temperature and velocity	• Particle trajectory • Particle temperature • Particle velocity • Particle distribution	• Coatings properties • Porosity • Mechanical properties • Deposition efficiency
	• Torch set up	• Atmosphere • Pressure Humidity		

Table 1.
The main parameters and characteristics of the APS process [15].

5. Atmospheric plasma spray micro-to-nano-sized structures

The applications of atmospheric plasma spray technology have changed considerably since its beginning in the 1950s. The global pressures on prices have forced companies to face challenges in their manufacturing processes; they generally answer by an acceleration of production, increasing in throughput and consistency in quality of the coating. Also, plasma-sprayed coatings have to cover a greater demand of applications such as higher operation temperatures, wear and corrosion under extreme conditions, and longer life span of parts and devices. A potential response for coatings with improved properties is the deposition of coatings with finer microstructure, i.e., finer lamellae and smaller voids as well as coating with microstructure more resistant to mechanical and thermal stresses than the lamellar microstructure exhibited by conventional plasma-sprayed coatings. This requirement has led to the development of innovative plasma coating processes for producing coatings with grain size in the nanometer range while keeping the high deposition rate and flexibility of plasma spraying [27, 28]. The process uses the basic equipment of the conventional plasma spray process but the feedstock is a liquid suspension or a solution of chemical precursors instead of the conventional powder feedstock, which takes advantage of the high enthalpy content of the plasma jet to evaporate the spray material and then forms a coating by fine droplets and/or condensation of the vaporized material on the substrate [29, 30]. To form the coating with liquid precursors, these must be injected into the plasma region in smaller droplets (sprayed or atomized). Then the solvent is evaporated, forming the solid material, which is melted (or forms a shell) and accelerated towards the substrate, as shown in **Figure 6**. As all these steps occur almost instantaneously, the plasma generator must provide suitable energy to process the liquid precursor and form the material.

Available since the 1990s, nanostructured materials are still considered a new concept that increase the performance of engineering components. Many studies have instigated the properties of nanostructured materials used in structural components and coatings. Ceramic materials gained attention mainly because they have greater hardness due to the smaller grain size, greater resistance to wear, and less incidence of defects [10, 31].

Nanostructured coatings can be obtained by plasma spray processes using liquid precursors. Solid precursors (powders) with nanometric distribution have difficulty with fluidity between the feed line and the plasma torch, causing intermittent injection of the material. The fluidity problem is overcome by increasing the flow of the carrier gas; however, this causes the plasma jet axis to shift and produce a non-uniform coating on the substrate. Another factor is that the nanoparticle may not penetrate the center of the plasma jet by inhibiting fusion and acceleration processes towards the substrate to form the coating [5, 32–35].

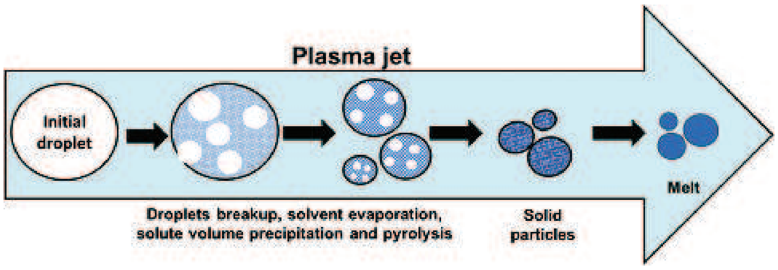


Figure 6.
Formation of the material from a liquid precursor with low and high concentration of the material to be deposited.

The solution plasma spray technique allows the deposition of thick nanostructured coatings (**Figure 7**), without the need for a very sophisticated infrastructure. Expenditure on materials (liquid precursor) is much lower than on the powders. The flexibility to use different feedstock enables a variety of compositions of the liquid precursor to be exploited by adjusting its concentration according to the desired application.

Biomaterials can be obtained with different techniques, thermal spraying shows significant advantages; in particular, the fact that the deposition and consolidation of the coating occur simultaneously without the need of a sintering treatment. Bioactive glasses are considered promising materials to be used as coatings onto implant devices, due to their high bioactivity [36, 37]. The use of solutions precursor instead of traditional thermal spraying feedstock provides unique properties, i.e., high purity materials (avoiding possible contamination from feedstock preparation steps), and nanostructured coatings with denser and more homogeneous microstructures [38].

Several efforts to use solution plasma spray process to fabricate superhydrophobic coatings have been reported [39]. Metals and metal oxides, as the most important and commonly used engineering materials, are hydrophilic for most part due to their high surface energy. There is immense interest in developing the ability to control the surface wettability of metals and metal oxides in order to improve their performance in corrosion resistance, friction reduction and efficiency in liquid transportation [40]. Xu et al. [41] presented superhydrophobic ceramic coatings with nano-sized hierarchical structure and high water contact angle, coatings were fabricated by a one-step solution precursor plasma spray process.

In addition, plasma reactivity can be exploited to obtain a final coating with composition different from the original precursor. In the study presented by Miranda et al. [12], a plasma spray system with axial injection was used to deposit nanostructured coatings. Coatings with graded composition between SiO_2/SiC (**Figure 8**) on carbon/carbon composites substrates were obtained.

A relevant result, obtained during the analysis of the chemical and structural composition of the coating, was the SiC formation due to the reactions between the carbon and the liquid precursor (SiO) promoted by the plasma jet. These reactions are exemplified by means of **Figure 9**. The formation of SiC in the coating helps to protect the substrate because it reduces the permeability of oxygen, preventing its oxidation and, consequently, the loss of its structural characteristics. Although no coating adhesion tests were carried out, the higher SiC concentration at the substrate/coating interface indicates the occurrence of a chemical adhesion process of the coating [42]. Thus, at this stage of the deposition process, the composite is

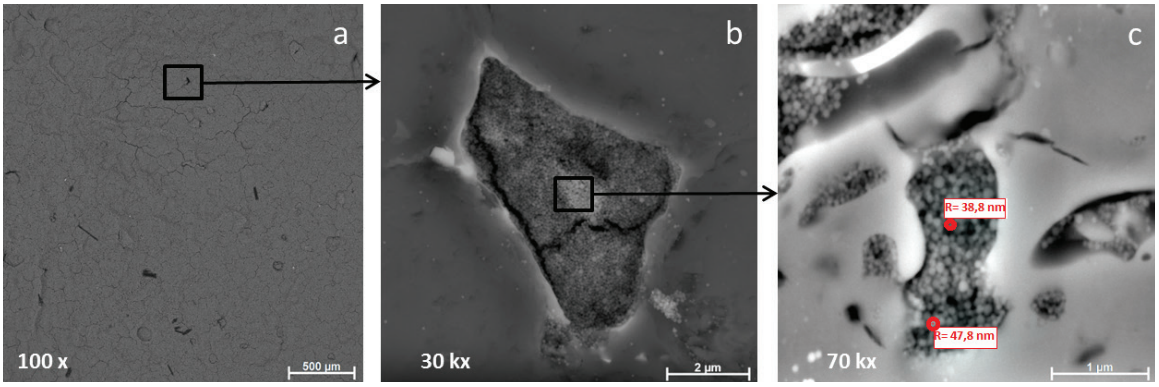


Figure 7.
(a) Top view of the coating, (b) nanostructure of coatings, and (c) particle measurements.

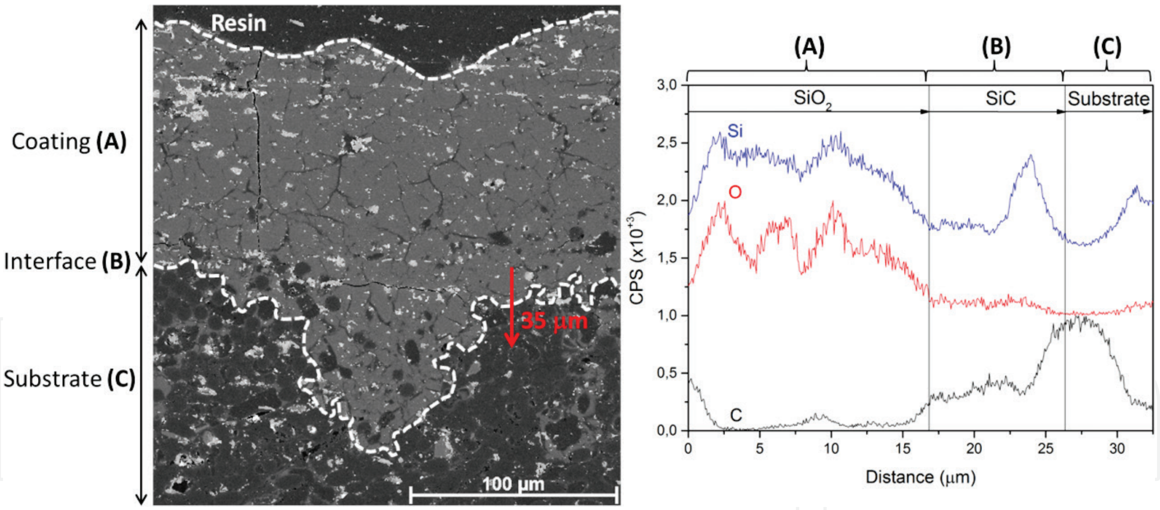


Figure 8.
SEM image of cross-section of sample and EDS results showing the composition of coating (A), interface (B), and substrate (C).

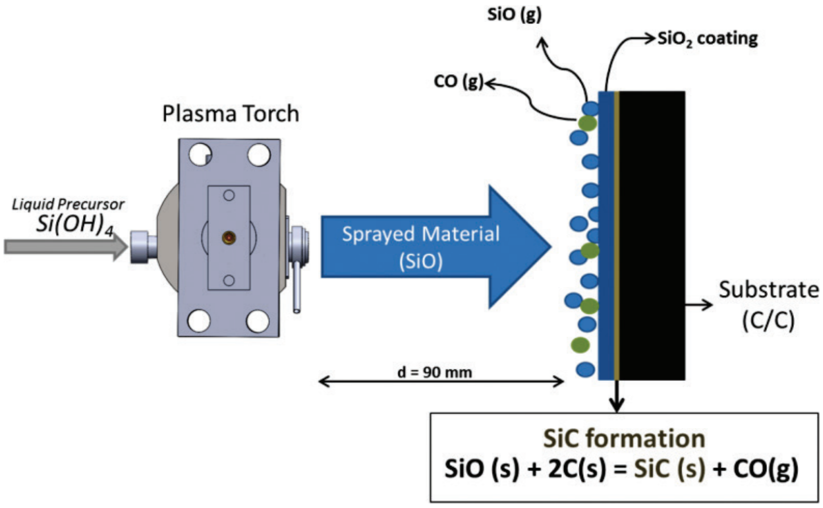


Figure 9.
SiC formation in the deposition process [12].

"doped" with a SiC layer and thus exposed to differentiated oxidation processes in relation to the original C/C substrate.

The coatings aim at the environmental protection of carbon/carbon composite substrates of great application in the aerospace sector. The main tool of this system is the non-transferred arc plasma torch calibrated and specifically characterized by forming a high enthalpy and high velocity plasma jet, capable to processing precursors with high melt temperatures at atmospheric pressure. With these characteristics, adjusted to power systems, gas feed and liquid precursors, it is possible to obtain nanostructured coatings with low fraction of pores and inclusions.

In processes using DC or RF plasma torches, typically the material is introduced into the plasma jet in the form of micrometric powders. They are accelerated due to moment transfer of the plasma jet and at the same time the process of heat transfer and mass begins. The residence time of the powder in the plasma jet, and consequently the efficiency of the process, depend on the particle's speed and the flight distance. The process of forming nanostructured coatings relates to these factors, since particles within the plasma flame must remain a sufficient time for total evaporation of the liquid precursor and melt the particles or particle's surface. The formation of nanostructures occurs with the rapid cooling of the vapor.

It is important to point out that this process is most commonly observed in works that employ short-arc plasma torches transferred for better utilization of process energy [43, 44]. In these systems, the vapor cooling process is the main stage of nanoparticle formation. Homogeneous nucleation is facilitated by the combination of heating, evaporation, and rapid cooling, such as happens in a plasma reactor [45].

6. Conclusions

Nanostructured materials have unique characteristics in relation to their mechanical and thermal properties, in addition to producing a lower index of defects and a lower porosity than coatings obtained in the traditional method using solid feedstock (which are limited to the size of the particle in flight). The use of atmospheric spray plasma systems is shown as an effective alternative to produce nanostructured and composite materials, which can be adjusted according to the need for application, as in the case of the use of liquid precursors. Therefore, it is necessary to advance the development of plasma torches capable of processing various types of materials to increase its range of application and consequently contribute to the technological advancement of materials processing.

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