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► **To cite this version:**

Alice Dolmaire, Simon Goutier, Aurélien Joulia, Pierre-Marie Geffroy, Michel Vardelle, et al.. Experimental Study of the Impact of Substrate Shape and Tilting on Particle Velocity in Suspension Plasma Spraying. *Journal of Thermal Spray Technology*, 2020. hal-02453433

**HAL Id: hal-02453433**

**<https://unilim.hal.science/hal-02453433>**

Submitted on 11 Dec 2020

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1 **Experimental study of the impact of substrate shape and tilting on particle velocity in**  
2 **Suspension Plasma Spraying**

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8  
9 **Abstract**

10 Suspension plasma spraying has shown its capacity to deposit finely structured  
11 coatings with a wide range of microstructures including columnar microstructures that are  
12 generally sought in thermal barrier coating applications for gas turbines. However, some  
13 challenges are still to be taken up before the application of the technology at an industrial  
14 scale. One deals with the deposition of a uniform and reliable coating on a complex shape  
15 substrate. This work offers an experimental observation of submicron particle streams close to  
16 the substrate in order to understand mechanisms of deposition. Effects of the substrate shape  
17 and tilting were investigated on particle velocity, directions and coating growth. It was shown  
18 that particle velocities and directions are disrupted by the substrate presence up to 10mm  
19 upstream. When the substrate is a cylinder or in a tilted orientation to the plasma jet, particles  
20 kinetic behaviour is less affected. Finally, submicron particle velocity vectors orientation near  
21 impact greatly shape the coating morphology. When impacting with a 40° angle of incidence,  
22 columns appeared on beads, contrary to submicron particle streams impacting orthogonally to  
23 the substrate surface.

1 **Keywords** particle velocity, suspension plasma spraying, thermal barrier coating, columnar  
2 coating, substrate shape, substrate orientation, PIV

3

4

## 5 **Introduction**

6       Suspension Plasma Spraying (SPS) is now emerging at the industrial scale, with most  
7 activities affecting new processes on the improvement of coatings on gas turbines. In this  
8 context, coatings need to be homogeneously deposited along the surface of turbine blades to  
9 be truly effective. However, turbine blades are made of complex forms with a variety of  
10 thicknesses, curves and type of edges, which greatly influence the morphology and quality of  
11 the coatings obtained by SPS [1]. To improve deposition rate and quality, further  
12 investigations are needed to understand the behaviour of submicron particles flow in the  
13 plasma jet impinging the surface of substrates.

14       In Atmospheric Plasma Spraying (APS), particles launched at high speed have  
15 trajectories quasi parallel to the gun axis and almost all resulting impacts on the substrate are  
16 orthogonal to the target surface [2,3]. The kinematic treatment depends a lot on particle mean  
17 size  $d_p$  which is in this case about more than  $10\mu\text{m}$  [2,4,5].

18       However, in Suspension Plasma Spraying (SPS), submicron and nano-sized particle  
19 trajectories are greatly affected by flow fluctuations [6] and any velocity gradients [5,7].  
20 Especially, near the target, these particles are very sensitive to the plasma flow directional  
21 change induced by the stagnation region [8], seemingly following plasma flow streamlines  
22 due to their very low Stokes number [9–12].

1           Moreover Pourang et al. [13], have numerically simulated the trajectory of a  
2 suspension of zirconia droplets within a plasma jet near a flat or curved surface. Their  
3 simulation has shown a significant influence of the substrate shape on particle trajectories in  
4 the vicinity of the substrate surface. Particles within a plasma jet impinging a cylindrical  
5 substrate were twice as likely not to deposit on the substrate surface. When impacting the  
6 cylinder, these particles also had a lower normal velocity (as regards the substrate normal  
7 axis) than particles impinging a flat substrate due to a narrower stagnation area.

8           Finally several models on coating growth in SPS have been suggested from  
9 experimental observations of coating morphologies obtained with varying sets of process  
10 parameters [14–18]. These models all agree on the great influence of particle direction and  
11 velocity when impacting the substrate on the resulting coating morphology. More precisely,  
12 the impacting directions of these particles on a peak of roughness seem to shape the coating  
13 into columnar morphologies via the shadowing effect.

14           Thus the presence of an obstacle totally disrupts the plasma and particle flow streams  
15 and affects their respective average velocity [19]. If submicron particles precisely follow  
16 plasma flow streamlines, as shown in simulations, how then is a SPS coating built when the  
17 centerline of the particle flow bearing a maximum of particles concentration rapidly  
18 decelerates and is easily deviated by the stagnation area?

19           No experimental investigation has yet been published, to the authors' knowledge, on  
20 the kinetic behaviour of suspension submicron particles in plasma jets impinging substrates.  
21 The objective of this study is therefore to provide it. Particle Image Velocimetry (PIV) will be  
22 used in order to observe submicron particles kinetic behaviour near two types of substrates, a  
23 flat substrate or a cylindrical one, with a tilted orientation or not to the plasma flow axis. Next  
24 the resulting coating morphologies will be analysed thanks to observations of cross-sections.

1

2

### 3 **Experimental methods and set-up**

#### 4 *Particle Image Velocimetry (PIV) principle and set-up*

5 PIV measurements consist in capturing movements of particles in a fluid thanks to an  
6 advanced optical set-up combined with a digital image correlation technique.

7 In this system (Fig. 1), two high-speed cameras were orthogonally placed to each  
8 other. The light is collected by the two cameras thanks to a beam-splitter. The lens of the first  
9 camera also determined the field of interest, i.e the spatial resolution. In this study the field  
10 was 12x8mm<sup>2</sup> and was 1mm deep. A double-pulsed Nd:YAG laser (wavelength  $\lambda=532\text{nm}$ )  
11 was used to radiate particles travelling through this field. The two cameras were then  
12 respectively synchronised to be exposed to only one pulse of the laser at a time in order to  
13 detect any movement in a couple of frames.

14 For this study, 100 image couples of dimensions 12x8mm<sup>2</sup> and of exposure time 1 $\mu\text{s}$   
15 were taken per operating condition. These conditions consisted in varying the substrate shape  
16 in front of the plasma jet as well as the tilting of the plasma gun in front of a flat substrate.  
17 The plasma gun working conditions were set constant during these sprayings. They are  
18 described in the next paragraph. Velocity fields were calculated from these 100 couples via an  
19 Eulerian approach thanks to the PIV software DaVis 8 (LaVision, Göttingen, Germany).  
20 Spatial auto-correlation was used to calculate the average pixel displacement of clouds of  
21 zirconia particles in an interrogation area of 128x128pixels within a partial window of  
22 dimensions 3x3mm<sup>2</sup>. An overlap of 50% was applied between each interrogation area. These  
23 correlation parameters allows to obtain up to 9000 velocity vectors for 100 velocity fields of  
24 dimensions 3x3mm<sup>2</sup>. Arithmetic averages of magnitudes of these velocity vectors were then

1 extracted from each windows of 3x3mm<sup>2</sup>. An average measurement error of 30m·s<sup>-1</sup> on  
2 particle average velocity was estimated from the slight variations of substrates positioning  
3 between and during spraying.

4

#### 5 *Spraying conditions and beads production*

6 The ethanol-based suspension was homemade using an 8wt.% yttria stabilized zirconia  
7 submicron powder ( $d_{50,v} = 0.7\mu\text{m}$ ) from IMERYS Fused Minerals (Laufenburg, Germany)  
8 and 2wt.% of phosphate ester as the dispersing agent (3DCeram, Limoges, France).

9 A TriplexPro-200 plasma gun from Oerlikon-Metco (Kelsterbach, Germany) was used  
10 to conduct the study. Spraying conditions are detailed in Table 1.

11 The suspension was then inserted in a pressurized tank with a magnetic agitation put  
12 underneath. It was later injected radially to the plasma jet at 4mm from the nozzle exit. The  
13 injector diameter was 150 $\mu\text{m}$  and the suspension flowrate around 30mL·min<sup>-1</sup>.

14 Two types of substrate were positioned at a standoff distance of 60mm (Fig. 2):

- 15 - A water-cooled circular copper plate, of diameter 50mm and thickness 28mm, put normal  
16 to the plasma flow in order to simulate a flat substrate (disk). It had a frontal area of  
17 1964mm<sup>2</sup>.
- 18 - Stainless steel rings, of diameter 50mm, height 32mm and thickness 2mm, set vertically  
19 to behave as cylindrical substrates with a curvature radius of 25mm and a frontal area of  
20 1600mm<sup>2</sup>.

21 In order to cool down substrate surfaces during each spray, standard TriplexPro  
22 commercial air-jets were used (details in Tab. 1). During PIV measurements, the cooled

1 copper plate was set immobile while stainless steel rings were mounted vertically on a chuck  
2 rotating at 100rpm (Fig. 3).

3 Part of the study is dedicated to apprehending the influence of the substrate orientation  
4 on the submicron particle flow behaviour. Due to the limitations of the PIV set-up, it was of  
5 best interest to orient the plasma jet with respect to the normal axis of the substrate instead.  
6 Indeed, this configuration allowed to recreate a plasma flow impinging an inclined surface as  
7 well as to maintain the same measuring volume. Two orientations of plasma jet were chosen:  
8 parallel to the normal axis of the substrate ( $0^\circ$  of incidence) or impinging with a  $40^\circ$  angle of  
9 incidence.

10 Next, this effect of the substrate orientation was studied by examining beads cross-  
11 sections. These beads were sprayed on grit-blasted stainless steel rings while maintaining the  
12 plasma gun motionless. Their cross-section were observed thanks to a JEOL IT300LV (Jeol  
13 Europe, Croissy sur Seine, France) scanning electron microscope in order to characterize the  
14 plasma jet position effect on the obtained coating morphology. Porosity in the beads was also  
15 evaluated from the micrographs by image analysis using the open-source software ImageJ.

16

### 17 *Estimated plasma jet thermophysical properties in the vicinity of substrates*

18 In order to interpret velocity results, thermophysical properties of the plasma jet were  
19 necessary for an 80%-20% argon-helium gas mixture with atmospheric air surrounding it at  
20  $x=60\text{mm}$ . Thus, the temperature, the viscosity and the density of the plasma flow were  
21 broadly estimated at  $x=60\text{mm}$  as follow.

22 The plasma temperature  $T_{\text{plasma}}$  was first estimated by measuring the melting point  
23 position  $T_{\text{alumina}}$  of an alumina rod of diameter 3mm in the plasma flow. The tip of the rod  
24 started to melt at  $x=55.5\text{mm}$  from the nozzle exit. Using previous work [20], values of the

1 heat transfer coefficient  $h$  were considered to vary between  $1500$  and  $3000\text{W}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$  and  
2 thermal flux density  $\Phi$  values in a range of  $2$  to  $3\text{MW}\cdot\text{m}^{-2}$  at  $x=60\text{mm}$  from the gun nozzle  
3 exit. Thermal gradient in the boundary layer of the plasma flow could then be evaluated  
4 thanks to the following equation (Eq. 1).

$$5 \quad \Phi = h (T_{\text{plasma}} - T_{\text{alumina}}) \quad (\text{Eq. 1})$$

6 Therefore, the plasma temperature  $T_{\text{plasma}}$  around  $60\text{mm}$  had been approximated to be  
7 roughly between  $5150$  and  $5850\text{K}$ . In order to simplify further calculations, plasma  
8 temperature was set on the average value, which was  $5500\text{K}$ . This value is close to previous  
9 SPS models with similar plasma conditions [11,13]. Thanks to this plasma temperature  
10 estimation, it was then possible to infer viscosity and density values for an argon-helium-air  
11 mixture at  $x=60\text{mm}$ . Volume fraction of air was set at  $90\%$  at this distance from the nozzle  
12 exit in the gas mixture [21]. Taking into account such air entrainment, plasma viscosity  $\mu_g$   
13 was estimated to be around  $1.6\cdot 10^{-4}\text{kg}\cdot\text{m}^{-1}\cdot\text{s}^{-1}$  and density  $\rho_g$  around  $5.4\cdot 10^{-2}\text{kg}\cdot\text{m}^{-3}$ . These  
14 calculated properties will be taken into account for the following estimations.

15

### 16 *Fluid dynamic of the plasma and particle flow in the vicinity of substrates*

17 Reynolds number of the plasma jet and drag forces applied on the substrates can be  
18 estimated at  $x=60\text{mm}$  thanks to the calculations in the previous paragraph. Submicron  
19 particles timely response to the plasma jet can also be gauged thanks to the Stokes number.  
20 These numbers and forces will help to better understand the dynamic behaviour of both flows  
21 (plasma and submicron particles) and thus will help into the interpretation of results on  
22 particle velocity.

23 Definitions of Reynolds number  $Re$ , drag force  $F_d$  and Stokes number  $St$  are described  
24 subsequently.



$$1 \quad \text{Re} = \frac{\rho_g v_g d_s}{\mu_g} \quad (\text{Eq. 2})$$

$$2 \quad F_d = \frac{1}{2} \rho_g v_g^2 A C_d \quad (\text{Eq. 3})$$

$$3 \quad \text{St} = \frac{1}{18} \frac{\rho_p d_p^2 v_g}{\mu_g D} \quad (\text{Eq. 4})$$

4            In the expression of the Reynolds number (Eq. 2), symbols  $\rho_g$ ,  $v_g$ , and  $\mu_g$  represent in  
5 this order density, velocity and viscosity of the mixture argon-helium-air at  $x=60\text{mm}$ . As  
6 zirconia particles suspended in the plasma jet in this study have a median diameter of  $0.7\mu\text{m}$ ,  
7 particle velocity is assumed to be equal to plasma velocity around  $x=60\text{mm}$  [5,11,13]. The  
8 average particle velocity value in a free jet at the centerline will be taken in this case. Symbol  
9  $d_s$  in this expression corresponds to the diameter of both substrates which is  $50\text{mm}$ .

10            Symbol  $A$  in the third equation represents the frontal area of the substrate, of value  
11  $1964\text{mm}^2$  for the flat substrate or  $1600\text{mm}^2$  for the cylindrical substrate.  $C_d$  is the drag  
12 coefficient of the substrates, depending on the Reynolds number of the plasma and air  
13 mixture.

14            In the Stokes number definition (Eq. 4),  $\rho_p$  and  $d_p$  are respectively density and mean  
15 diameter of the submicron zirconia particles, with respective values of  $5890\text{kg}\cdot\text{m}^{-3}$  and  $0.7\mu\text{m}$ .

16            Then in a similar fashion than Anderson and Longmire [22], two Stokes numbers will  
17 be computed,  $\text{St}_0$  corresponding to the particle-fluid relation at the nozzle exit of the plasma  
18 gun ( $\text{St}_0 = \rho_p d_p^2 v_{g0} / 18 \mu_{g0} D_0$ ) and  $\text{St}_{60}$  corresponding to the particle-fluid relation far  
19 downstream, around  $60\text{mm}$  ( $\text{St}_{60} = \rho_p d_p^2 v_g / 18 \mu_g D_0$ ). In the first case, the plasma velocity  
20 at nozzle exit  $v_{g0}$ , the diameter of the nozzle  $D_0$  and the viscosity of the plasma mixture at  
21 nozzle exit  $\mu_{g0}$  are taken into account (values in Tab. 1).

22

1

## 2 **Results and discussions**

3           The aim of these measurements is to visualize the particle flow in the vicinity of the  
4 substrate surface and estimate the average value of the magnitude of particle velocity both  
5 axially, along the centerline of the plasma jet, and radially, along the plasma jet radius.

6

7 *Axial evolution of particle velocity at the center of the plasma jet, comparisons between free*  
8 *jets and impinging jets at  $x=60\text{mm}$  from the nozzle exit*

9           First, it is worth noting that the use of commercial cooling air-jets surrounding the gun  
10 nozzle had an impact on the plasma flow behaviour in front of the substrates. Indeed working  
11 with such cooling air flow macroscopically created an air barrier before the surface of the  
12 substrates compared to a spray with no air-jets in the same operating conditions (Fig. 4). This  
13 cold barrier had a thickness around 3mm according to the corresponding velocity fields.  
14 Despite these observations, cooling air-jets were maintained to a minimum value, for every  
15 tested operating condition in order to minimise experimental variables and optimise the  
16 survival of substrates during and after each spray.

17

### 18 **Effect of the presence of a wall on the axial evolution of particle velocity**

19           Average particle velocities were measured at the center of the plasma jet ( $y=0\text{mm}$ )  
20 between  $x=50\text{mm}$  and  $60\text{mm}$  from nozzle exit. As reported in Fig. 5, velocity measurements  
21 of suspensions in a free jet showed a natural deceleration of  $70\text{m}\cdot\text{s}^{-1}$  in  $10\text{mm}$ , from  $x=50\text{mm}$   
22 to  $60\text{mm}$ . On the other hand, free jet with surrounding air-jets showed a deceleration of about  
23  $100\text{m}\cdot\text{s}^{-1}$  on a distance of  $6\text{mm}$  from  $x=50.5\text{mm}$  to  $56.5\text{mm}$ . Additionally, these particles

1 travel faster than the particle jet impinging a surface positioned at  $x=60\text{mm}$ . Indeed, a  
2 maximum gap of  $200\text{m}\cdot\text{s}^{-1}$  at  $53.5\text{mm}$  from nozzle exit was registered between particles  
3 dragged in a free jet compared to particles encapsulated in a plasma jet impinging a flat  
4 substrate, with cooling air-jets in use in both cases.

5 Stokes numbers were computed for the submicron particle flow along the axis of the  
6 plasma jet and evolve from  $0.16$  ( $St_0$ ) to  $0.06$  ( $St_{60}$ ). In all cases, Stokes numbers are below 1.  
7 This means zirconia submicron particles are very sensitive to the plasma jet behaviour and  
8 strictly follow the plasma jet streamlines even with a decreasing plasma viscosity.

9 All of the above indicates the presence of a wall in front of the plasma jet disrupts  
10 greatly its dynamic behaviour which results in braking the suspension particle flow  
11 significantly upstream within the plasma jet. This braking effect is also an indirect indication  
12 of the existence of a stagnation area for the plasma flow created by the presence of a  
13 substrate. A stagnation area is a definite space where fluid particles are brought to rest due to  
14 a particular geometry of an obstacle met by the plasma flow. The dimensions of this flat  
15 substrate suffice in creating such an area.

16 Moreover the addition of said cooling jets has a negative effect on particle velocity.  
17 When comparing both particle flows impinging a flat substrate (Fig. 5), average velocity  
18 values drop off between  $100\text{m}\cdot\text{s}^{-1}$  and  $150\text{m}\cdot\text{s}^{-1}$  at respectively  $50.5\text{mm}$  and  $53.5\text{mm}$  from  
19 nozzle exit. Naturally this loss of speed depends a lot on air nozzle orientation and air flow  
20 rates. In many industrial cases implementing a rotating substrates carrier, it is then beneficial  
21 to turn off standard air-jets fixed on the gun, and cool down the substrates using others  
22 systems.

23 Thus, when spraying on a surface the average magnitude of particle velocity is  
24 affected by a double negative effect: the use of cooling air-jets surrounding the plasma jet and

1 the presence of the surface itself which disrupts and brakes the particle flow upstream, in this  
2 case up to 10mm upstream.

3

#### 4 **Effect of the shape of the substrate on the axial evolution of particle velocity**

5 As shown in Figure 6, the average particle velocity values did not change significantly  
6 in front of the cylindrical or flat substrate geometries used in this work. Moreover, particle  
7 velocities at the center of the plasma jet decrease at the same rate in both cases. This lack of a  
8 significant difference may be the result of the small and similar cross sectional shapes of the  
9 geometries chosen. Indeed, Reynolds number of the plasma flow in front of these substrates is  
10 identical (diameter is identical for both substrates) and of magnitude  $5 \cdot 10^3$ .

11 However, drag coefficients for these substrates at this value of Reynolds number  
12 depend on their aspect ratio according to Hoerner's experimental diagrams [23]. For the flat  
13 substrate, it is a disk of aspect ratio length/diameter of 0.56, therefore a drag coefficient  $C_d$  of  
14 1.02 according to Hoerner. In the case of the cylindrical substrate the aspect ratio  
15 diameter/length was of 1.56 which corresponds to a drag coefficient  $C_d$  tending to 0.70. Drag  
16 forces of these substrates are then respectively of 4.9N for the flat substrate and 2.7N for the  
17 cylindrical substrate. Therefore, the cylindrical substrate should show slightly less resistance  
18 to the plasma flow.

19 The braking effect on the particle velocity produced by the substrate appears on Fig. 6  
20 to extend less strongly upstream with a cylindrical surface than with a flat substrate, higher  
21 average velocities being registered in this former case. This trend is due to the curvature  
22 radius (25mm) of the cylinder which impacts the drag force on the plasma jet and is coherent  
23 to the drag force calculation which is lower for the curved substrate. Increasing the length or  
24 reducing the curvature radius of the cylinder would likely exacerbate any differences in the

1 particle flow kinetic behaviour in the vicinity of these two types of substrate. Moreover, the  
2 curvature radius of the cylinder implies that plasma jet streamlines along the z axis are less  
3 and less subjected to an obstacle orthogonal to their trajectories. Therefore, with this curved  
4 target the stagnation zone is also weakened in size and submicron particles are less  
5 decelerated by this change of plasma flow behaviour. This trend on velocity results in front of  
6 a curved substrate is in agreement with simulations [13, 19].

7

8 *Radial evolution of particle velocity in a free jet case or in the presence of substrates at*  
9 *x=60mm from nozzle exit*

10 To further this investigation, velocities were measured along the radius of the plasma  
11 jet, which corresponds to the determination of velocity vectors along the y axis. Figure 7  
12 shows average values of magnitude of these velocity vectors at x=50.5mm and 56.5mm from  
13 nozzle exit.

14

### 15 **Free jet case**

16 As in most cases in thermal spraying, in both cross-sections, x=50.5mm and 56.5mm,  
17 the particle velocity radial distributions (Fig. 7) are symmetrical and show a maximum at the  
18 center of the free plasma jet. It noted that the Gaussian evolution ordinary observed for a jet is  
19 not very steepened in both cross-sections, with an average value of  $315\text{m}\cdot\text{s}^{-1}$  at 50.5mm and of  
20  $230\text{m}\cdot\text{s}^{-1}$  at 56.5mm in a spot of 8mm in diameter.

21

### 22 **Jet impinging a flat substrate**

1            In the case of a plasma jet impinging the flat substrate, some changes are noticed  
2 compared with the kinetic behaviour in a free jet case (Fig. 7). First, at  $x=50.5\text{mm}$ , every  
3 velocity value along the  $y$  axis is about  $100\text{m}\cdot\text{s}^{-1}$  lower than velocity values registered in the  
4 free jet case in the same spatial position. Secondly, the concave shape of velocities  
5 distribution along the  $y$  axis flattens and becomes a rather convex curve at  $x=56.5\text{mm}$  from  
6 the nozzle exit. At  $3.5\text{mm}$  upstream of the surface of the flat substrate, the lowest velocity  
7 value  $74\text{m}\cdot\text{s}^{-1}$  is then at the center of the plasma jet.

8            These evolutions are mainly due to a maximum of static pressure in the center of the  
9 plasma jet when meeting a wall, thus due to the effect of a stagnation zone on the plasma  
10 flow. These velocity results shows that the stagnation zone is located especially in the center  
11 of the plasma jet with a decreasing intensity on static pressure along the radial axis in just a  
12 few millimetres. The plasma flow must then be deviating in a wall jet configuration in order  
13 to explain the higher velocity values of particles registered around this center.

14

### 15            **Jet impinging a cylindrical substrate**

16            When comparing the radial evolution of velocities between the submicron particle  
17 flow impinging a cylindrical substrate and a flat substrate (Fig. 7), the convex shape of the  
18 velocity curve already appears at  $x=50.5\text{mm}$  and is preserved along the way. However,  
19 velocities are higher for a flow impinging the cylindrical substrate than a flow impinging the  
20 flat substrate, which can be easily explained by the drag forces. At  $x=50.5\text{mm}$ , the particle  
21 velocity radial evolution is even almost identical in magnitude to the free jet except for the  
22 velocity value at the centerline. This shows the stagnation zone is radially narrower in size  
23 and extends less farther along the plasma flow axis with this type of substrate than with the  
24 flat substrate. The additional presence of air cooling flows may also provide an air

1 acceleration around the substrate to the more peripheral particles and emphasize this curvature  
2 effect. Consequently, the flow kinetic behaviour is also less affected radially by the presence  
3 of a substrate with such a curvature (25mm) than when it is impinging a flat surface.

4

5 *Tilting effect of the plasma gun on particle flow kinetic behaviour near the substrate and*  
6 *beads morphology*

7

### 8 **Tilting effect of the plasma gun particle velocity near the flat substrate**

9 The angle between the normal axis of the substrate surface and the gun axial axis was  
10 set at 40°.

11 Figure 8 shows two instantaneous particle velocity fields between the two cases as  
12 well as the resulting average value at 3.5mm away from the substrate. This average value was  
13 obtained from the summation of all 100 velocity fields contained in a measurement window  
14 of 3x3mm<sup>2</sup> and centered at 56.5mm. Respectively, an average value of 74m·s<sup>-1</sup> was measured  
15 when the plasma jet is impinging the surface in a parallel direction of the normal axis, i.e with  
16 0° of incidence to the normal axis, and 143m·s<sup>-1</sup> was registered when the gun was set at a 40°  
17 angle from the substrate normal axis.

18 Moreover, the instantaneous particle velocity fields display that clouds of submicron  
19 particles follow the plasma jet general direction. Indeed, vectors are preserving an incident  
20 angle of approximately 0° or 40° to at least 5mm from the surface of the substrate. In the  
21 case of an incidence angle close to 0°, vectors around the centerline are starting to diverge  
22 outward and symmetrically at 5mm with a gradually decreasing magnitude. When the  
23 incidence angle is 40° to the normal axis, most of the vectors in the y- area are diverging

1 away from the surface of the substrate, starting 12mm upstream. In the  $y^+$  area, vectors  
2 divergence only takes place starting 5mm upstream the surface with an opening angle of  
3 about  $45^\circ$  to the substrate normal axis at  $y=0$ . Their divergence away from the substrate  
4 surface also keeps the set direction towards the  $y^-$ . No vector was registered going against this  
5 general downward direction within the limits of the spatial dimensions of these images. The  
6 magnitude of the velocity vectors at the centerline of the plasma jet, thus diagonally on the  
7 instantaneous field (Fig. 8), also tends to decrease way less until impact compared to the  
8 impinging jet with a  $0^\circ$  incidence, hence the overall greater average velocity value registered  
9 in this area.

10 Thus, enough tilt of the substrate leads to submicron particles being less decelerated  
11 upon impact although their flow direction is more affected by it as well. The orientation of the  
12 tilt leads to a preferred general direction of the submicron particle flow with the same  
13 orientation. It also means there is a less impactful stagnation zone to the plasma flow, in terms  
14 of geometrical dimensions.

15

## 16 **Beads morphology**

17 Next, the influence of the angle of incidence of the general submicron particle flow  
18 was studied on beads. Figure 9 displays SEM images of the beads morphologies in relation  
19 with the orientation of the plasma jet to the normal axis of the substrate.

20 These morphologies correlate strongly with the registered particle flow direction  
21 before the substrate. Beads produced with a gun placed orthogonally to the substrate, thus  
22 with a incidence angle close to  $0^\circ$  at the centerline, display a homogeneous and rather dense  
23 morphology. Beads produced with a tilted plasma gun exhibit a morphology composed of  
24 columns with a strong directional growth oriented toward the center of the plasma jet. The



1 angle described by the columns orientation and the normal axis of the substrate is of about 30°  
2 regardless of the position in the coating from the center of the plasma jet.

3 The noticed difference between this column growth angle and the incidence angle of  
4 the particles impacting has been similarly reported before on columnar microstructures  
5 produced by physical vapour-phase deposition techniques, such as PS-PVD [24] and PVD  
6 [25]. These authors seem to agree that this difference is due to atomistic particles self-  
7 shadowing while the coating is growing. A similar phenomenon of particles self-shadowing  
8 could likely be happening with these submicron particles, leading to columns with an  
9 orientation towards the center of the particle flow and with a lesser angle of growth than the  
10 incidence angle of the particle flow.

11 Porosity at the center of the beads was also evaluated thanks to image analysis. In both  
12 beads porosity was around 20%. Therefore it seems that neither impact velocity nor jet  
13 orientation at the centerline of the flow influence drastically the porosity level within the  
14 coating. Orientation of the plasma jet and impact velocities only influence the stacking of  
15 splats into columns or layers.

16

17

## 18 **Conclusions**

19 This study leads to a better understanding of coating growth by SPS thanks to the  
20 observation of the submicron particle velocity and direction in a plasma jet impinging on  
21 different types of substrate. Due to the very small particle sizes, a PIV set-up had to be used to  
22 apprehend this flow. Different substrates were then chosen: flat, curved or inclined.

23 It has been shown that:

- 1 - The submicron particle flow consistently follows the plasma jet streamlines.
- 2 - The substrate presence disrupts the particle flow direction and velocity up to 10mm  
3 upstream from the substrate surface in comparison with free jets.
- 4 - The positioning of air-jets is of major importance as the air flow can provoke a decrease  
5 of the particle flow average velocity near the substrate.
- 6 - The substrate shape has an impact on the particle flow kinetic behaviour. Particles  
7 impinged the cylindrical substrate (curvature radius of 25mm) with a higher velocity than  
8 particles impinging the flat substrate.
- 9 - When trapped in a plasma jet impinging the flat substrate with an incidence angle of  $40^\circ$ ,  
10 the particle flow keeps the plasma jet incidence at the centerline and is then less deviated  
11 and slowed down upon impact.
- 12 - The incidence angle of the submicron particle flow had a great impact on the resulting  
13 columns orientation of the coatings and on their deposition homogeneity.

14         These experimental results also highlight the existence of a stagnation zone to the  
15 plasma jet flow and particle flow in the SPS process.

16         However, a better characterization of the submicron particle direction and velocity  
17 upon impact is still required in order to attain a better understanding of the effect of the  
18 stagnation zone on coating growth in SPS. The next study will thus focus on better describing  
19 submicron particles kinetic behaviour very near a flat substrate in order to measure impacting  
20 velocities and their incidence angles.

21

22

23 **Acknowledgements**

1           The authors would like to thank the French National Association of R&T (ANRT)  
2 for the financial support.

3

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- 1 **Fig. 1** Diagram of the PIV set-up used in this study
- 2 **Fig. 2** Diagram representing the spraying distance and substrates in use
- 3 **Fig. 3** Photography of a spray during PIV measurements done in presence of a rotating  
4 stainless steel ring and cooling air-jets
- 5 **Fig. 4** Photography of two spray in front of a stainless steel ring with air-jets off or air-jets on
- 6 **Fig. 5** Evolution at the centerline of the plasma flow of particle average velocity magnitudes  
7  $|v|$  with or without cooling air-jets or with or without the substrate presence at  $x=60\text{mm}$  from  
8 plasma gun nozzle exit
- 9 **Fig. 6** Average of particle velocity magnitudes at the plasma jet centerline when impinging  
10 cylindrical and flat substrates positioned at  $x=60\text{mm}$  and with the use of air-jets
- 11 **Fig. 7** Radial evolution of the average of magnitudes of particle velocities at  $x=50.5\text{mm}$  or  
12  $x=56.5\text{mm}$  from nozzle exit, in the case of a free plasma jet and in the presence of a  
13 cylindrical or flat substrate situated at  $x=60\text{mm}$
- 14 **Fig. 8** Tilting effect of the plasma gun on particle velocity at  $3.5\text{mm}$  from the flat substrate  
15 (window of measurement represented by the dotted rectangle) and on instantaneous particle  
16 velocity fields. Green arrows help to illustrate velocity vector directions & magnitudes  
17 represented by smaller red arrows. The white clouds are puff of zirconia particles impinging  
18 the surface
- 19 **Fig. 9** SEM images of beads obtained on rotating cylinders with a  $0^\circ$  (a) or  $40^\circ$  angle of  
20 incidence of the plasma jet (b) to the substrate normal axis. A sketch in (b) also displays the  
21 angle of columns to the substrate normal axis

22

1 **Table 1** Spraying conditions

Gas mixture Ar/He	80%/20%
Total gas flow rate	50L·min <sup>-1</sup>
Nozzle diameter D <sub>0</sub>	6.5mm
Electric power	23.5kW
Plasma jet mass enthalpy	22.2MJ·kg <sup>-1</sup>
Plasma jet temperature (at nozzle exit, estimated from mass enthalpy)	13400K
Plasma jet velocity v <sub>g0</sub> (calculated average at nozzle exit)	1450m·s <sup>-1</sup>
Plasma jet viscosity μ <sub>g0</sub> (estimated from mass enthalpy)	2.30·10 <sup>-4</sup> kg·m <sup>-1</sup> ·s <sup>-1</sup>
Stand-off distance	60mm
Injector diameter	150μm
Suspension flow rate	30mL·min <sup>-1</sup>
Air-jets flow rate	15m <sup>3</sup> ·h <sup>-1</sup>
Chuck rotation speed	100rpm

2