Measurement of the in-flight particle characteristics in plasma spraying by phase Doppler anemometry (PDA)

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**Measurement of the In-flight Particle Characteristics
in Plasma Spraying by Phase Doppler Anemometry
(PDA)**

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Abstract

Phase Doppler Anemometry (PDA), as a single-point optical measurement technique with unique capability of simultaneous measurement of the single spherical particle velocity, diameter and even volume flux, has demonstrated its superior advantage in characterizing many polydisperse flows. However, its application in measuring the plasma sprayed particles is relatively new, and specific considerations for applying the PDA technique in the plasma spraying field as well as the specific data interpretation requirement have never been mentioned before. In the present thesis, emphasis was first put on investigating the accuracy of the PDA measured particle diameter and the volume flux. The PDA diameter measurement was tested for different powder groups with known size range. The evaluation was further supplemented by the comparison of the PDA measured particle volume flux with the volume of the actual deposit. It was found that some particles passing the diameter validation criteria of the PDA could actually be recorded with miscalculated diameters by the PDA software. Their existence, especially for the smaller particles which were mistakenly calculated as larger ones, would significantly affect the particle volume flux distribution. However, the modified volume flux distribution obtained by filtering these larger miscalculated particles exhibited a close surface profile and total volume value with those of the corresponding actual deposit. The variation of the local thickness distribution and the total volume of the deposit with the plasma spraying parameters could also be approximately estimated by the PDA modified particle volume flux distribution.

Abstract

With the reliability and the error reduction method of the PDA measurement recognized, the PDA technique was then utilized to characterize the in-flight behaviors of the plasma sprayed particles under different spraying conditions. The 8wt.% yttria partially stabilized zirconia (YSZ) powders were atmospherically plasma sprayed from a Miller SG-100 plasma torch by systematically varying the primary four user adjustable parameters, i.e. the plasma gas flow rate, carrier gas flow rate, arc current level and powder feed rate. The PDA measurements were carried out along the radial direction at a fixed standoff distance of 80mm from the nozzle exit for the powder injected through the conventional one transversal injection port and a novel opposite two injection ports, both located at either upstream (internal) or downstream (external) of the plasma torch exit. The reason for introducing the two-port powder injection scheme was that both the in-flight particle distribution and the coating formed under this condition would be expected to be more uniform. Corresponding to some typical operation conditions for the PDA measurement, coatings/deposits were sprayed onto sand-blasted mild steel substrates for the microstructure and deposition efficiency determination. Through the comparison of the in-flight particle characteristics and the off-line coating/deposit analysis, it was concluded that the particle temperature at impact had the most predominant influence on the coating microstructure and the deposition efficiency. In addition, efforts were devoted for the first time to studying and establishing a correlation of the coating microstructure and deposition efficiency with the PDA measured particle volume flux. The impetus for doing so was that successful predicting the variation of the coating properties by on-line monitoring of the particle in-flight characteristics would possess superior advantage over the traditional way of off-line measurements, which could be very time-consuming and less reliable. The legitimacy of relating the coating deposition

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efficiency to the PDA measured particle concentration and volume flux was based on the fact that both of them were sensitive to and would change with the amount of melted or surface-melted particles at impact. Through the investigations, it was found that the PDA measured radial distribution of the particle volume flux could quantitatively reflect the variation of the coating microstructure and deposition efficiency. Although it was unanimously agreed that the particle velocity and temperature at impact were the most important parameters determining the coating properties, their influences on the coating properties were not straightforward and the prediction of the coating properties based on them were usually indirect and unreliable. The particles validated for the volume flux measurements were most probably in the molten or surface-melted state, and it were just these particles that had contributed most to the actual coating/deposit formation. Therefore, the variation of the PDA measured particle concentration and volume flux was consistent with that of the coating properties, and they served as the most direct link to assess quantitatively the coating microstructure and the deposition efficiency.

Keywords: Plasma spray; Yttria partially stabilized zirconia (YSZ); Phase Doppler Anemometry (PDA); Volume flux; Coating microstructure; Deposition efficiency

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List of Symbols

$A(d_p)$	Particle size-dependent effective cross-sectional area of the PDA measurement volume
A_p	Surface area of particle
C_N	Particle concentration
d_e	Initial laser beam diameter before exiting from the transmitter front lens
d_f	Laser beam waist diameter
d_p	Particle diameter
d_x	Height of the PDA measurement volume
d_y	Width of the PDA measurement volume
d_z	Length of the PDA measurement volume
D_{30}	Particle mean volume diameter
f_0	Fixed frequency shift
f_D^*	Doppler frequency
f_r	Focal length of receiver
f_t	Focal length of transmitter
F	Powder feed rate
F_D	Drag force
F_N	Particle volume flux
G_d	Weight gain of the deposit
l_s	Width of slit
L_d	Length of the deposit stripe
L_s	The apparent slit width
m	Relative refractive index
m_p	Mass of the particle
n_m	Refractive index of medium
n_p	Refractive index of particle
N_f	Total number of fringes

List of Symbols

p	Scattering mode parameter
$P(n)$	Possibility of n particles concurrence inside the PDA measurement volume
$r(d_p)$	Particle size-dependent effective cross-sectional width of the PDA measurement volume
Re	Reynolds number
u	Particle velocity along main flow direction
U	Plasma velocity
v	Particle velocity along powder injection port direction
V	PDA measurement volume
V_T	Traverse speed of the plasma torch
α	Off-axis angle or the PDA scattering angle
ψ	Elevation angle of the PDA receiver
θ	Laser beam intersection angle
γ	Particle trajectory angle
λ	Wavelength
μ_g	Dynamic viscosity of the plasma
η	Particle number density
β	Phase factor or geometrical factor
$\Delta\Phi$	Phase difference
ρ_g	Plasma density
ρ_p	Particle density
δ_f	Fringe spacing
τ	Angle between the particle surface tangent and the incident laser ray
τ'	Angle between the particle surface tangent and the refracted laser ray

Chapter 1

Introduction

1.1 Background

Plasma spraying is a well-established technique in producing various kinds of coatings for many engineering applications such as automotive, chemical, and aerospace industries [1,2]. As one of the many thermal spray processes including flame spraying, HVOF, detonation flame spraying, arc spraying etc., plasma spraying has demonstrated its superiority over others in many applications due to its ability to create a plasma jet with extremely high temperature and velocity. Many non-decomposing materials can be sprayed effectively under these conditions.

In order to understand plasma spraying, the term “plasma” must first be elucidated. Basically, a plasma consists of a mixture of electrons, ions, and neutrons in the ground state, excited species, and photons [1]. Such a mixture, however, qualifies as a plasma only if the negative and positive charges balance each other, i.e. overall a plasma must be electrically neutral. This property is known as quasi-neutrality. In contrast to an ordinary gas, plasmas are electrically conducting due to the presence of free charge carriers. In fact, plasmas may reach electrical conductivities exceeding those of metals at room temperature.

Plasma can be generated by passing a gas through an electric current. Since gases at room temperature are excellent insulators, a sufficient number of charge carriers must be generated to make the gas electrically conducting. This process is known as electrical

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breakdown, and there are many possible ways to accomplish this breakdown. Breakdown of the originally nonconducting gas establishes a conducting path (electric arc) between a pair of electrodes. For the plasma generated by a plasma torch, the electric arc is ignited between a thoriated tungsten cathode and a copper anode in the form of a nozzle surrounding the cathode. The plasma forming gases at present are mainly argon, helium, hydrogen, nitrogen or a combination of them. The plasma gas entering the nozzle is heated by the electric arc energy to a high temperature, causing the dissociation of the plasma gas. Through the dissociation and ionization processes, the plasma gas transfers into a plasma state, and a large volume of thermal and kinetic energy is released, which leaves the nozzle in the form of a plasma jet.

The plasma spray process encountered most frequently is the delivery of solid particles through a plasma torch [3-5]. Particles introduced into the plasma torch are melted and accelerated during the process of heat and momentum transfer from plasma to the particles in the plasma jet. The molten or partially molten particles are then propelled, in the form of a particle plume, towards the substrate to form the coating, as depicted in **Figure 1.1**). When the sprayed particles impinge upon the surface, they cool down and build up, splat by splat, into a laminated structure of plasma sprayed coating. **Figure 1.2** illustrates a typical coating cross section of the laminated structure with porosities, oxides and inclusions [6].

From the brief introduction mentioned above, it is obvious that there are generally three basic phases involved in the plasma spray process: plasma jet generation, plasma-particle interaction, and coating formation [7]. Each of these three sub-processes is actually influenced by a number of parameters, either controllable or uncontrollable. They would

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affect the final coating properties [8-10]. Due to the large temperature and velocity gradients existence in the plasma jet, small changes in some of the spraying parameters may result in significant variations to the coating properties [11].

In order to ensure the predictability and reproducibility of the plasma sprayed coating, proper control of the plasma spraying parameters is of utmost importance. Traditionally, the plasma spraying processes can be optimized and controlled by empirically tuning of numerous spray gun parameters and examining the individual properties of the resultant coating. This approach has major drawbacks, since too many parameters need to be monitored and controlled, and their influences on the coating properties are often interrelated and non-linear. In addition, other uncontrollable parameters such as the electrode wear and changes in the particle injection conditions make the relationship between the individual spraying parameters with the coating properties even more confusing and unpredictable [12].

The method that empirically examines the influence of individual plasma spraying parameters on the coating properties has been gradually discarded, replaced by the more scientific and thorough investigations on the fundamental mechanisms inherited in every aspect of the plasma spraying process with the aid of various sophisticated optical measurement devices [12, 13]. Investigations on the plasma jet generation emphasis plasma torch design and the influence of plasma parameters, for example arc power, the plasma gas composition, gas injection method, gas flow rate, surrounding atmospheric condition, chamber pressure and so on, on the plasma jet temperature and velocity distributions [14-16]. The instabilities of the plasma jet caused by the anodic arc root fluctuations, entraining of the cold ambient air into the plasma jet, and the wear of

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electrodes have also drawn great attention [17, 18]. As the medium for heating and accelerating the solid particles, any change of the plasma jet properties will naturally be reflected on the in-flight particle characteristics. The particle in-flight behaviors are not only influenced by the plasma parameters, but also by the way the particles are being injected inside the plasma jet [19]. For a given plasma jet, the momentum of the injected particles, which is primarily influenced by the injected particle velocity and size, has to be adapted to that of the plasma jet to enable the majority of injected particles travel along the hottest zone of the plasma jet. Particle trajectories depend on many factors: for example, the particle size and injection velocity, and the position and tilting angle of the powder injector [20]. Investigating directly on these in-flight particle behaviors may provide a better insight into the plasma-particle interaction. Furthermore, it has been demonstrated that the microstructure and properties of the plasma sprayed coating are closely related to the particle surface temperature and velocity upon impacting on the substrate, as well as the conditions of the substrate such as the pre-heating temperature and surface roughness [7, 21-22]. Thus, intensive efforts have been devoted to investigating the particle in-flight characteristics and the substrate conditions on the splat formation and the coating properties.

1.2 Objectives

The major object of this thesis is to provide a more convenient and reliable approach to determine the optimal setting of the plasma spraying parameters, which is usually hard to be ascertained when spraying different powder materials using different plasma spraying systems. The emphasis of the present research is on systematically investigating the influence of some typical user-adjustable plasma spraying parameters in one of the most popular commercial plasma spray systems, namely the SG-100 (Miller Thermal Inc. USA)

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plasma torch (**Figure 1.3**) controlled by the plasma control console (Praxair, Model 3702), on the in-flight particle characteristics for difference powder injection schemes with the aid of a Phase Doppler Anemometry (PDA) technique, and on the microstructure and deposition pattern of the coating/deposit formed within an Air Plasma Spraying (APS) environment. Efforts are devoted for the first time, according to the author's knowledge, to examining the accuracy and reliability of the PDA technique with respect to measuring the particle diameter and volume flux of the plasma sprayed powders (NiCrAlY and 8wt.% Yttria Stabilized Zirconia (YSZ) powders). With the level of accuracy of the PDA measurement in the plasma spraying process being identified, attentions are paid on using the PDA method to characterize the variation of the in-flight particle velocity, diameter, concentration and volume flux distributions under different spraying conditions, which included various settings of the plasma spraying parameters for powders injected through various injection methods, both internally and externally. Corresponding to some typical spraying conditions for the PDA measurement, coatings/deposits are sprayed onto the sand-blasted mild steel substrates for the microstructure and deposition efficiency determination. Through the comparison of the in-flight particle characteristics and the off-line coating/deposit analysis, attempts have been made to establish a correlation between them with the purpose of more accurately predicting the coating properties with the in-flight particle information.

1.3 Scope of Thesis

In **Chapter 2**, a systematical review will be presented addressing the most important diagnostic methods and results for the three basic phases of plasma spraying, i.e. plasma jet generation, plasma-particle interaction, and coating formation respectively. This review is

structured to provide the fundamentals and references for the investigations carried out in this thesis.

The experimental apparatus and the principle of the PDA will be described in **Chapter 3**. The particulars of the plasma spray system and powders employed in the present investigations will be introduced first, followed by the elucidation of the principle of the PDA technique with respect to simultaneous measurement of the particle velocity, diameter, concentration and volume flux. Since the optical alignment is critical for reliable measurement, the optimization of the important parameters of the PDA will be emphasized. The potential error sources inherited with the PDA measurements will also be analyzed and the general approaches for their minimization will be introduced.

It is well known that the successful application of the PDA technique relies on the physical properties of particles, flow conditions and the PDA configurations [23]. The reliability of the PDA measurement for characterizing a specific flow condition must be ascertained; otherwise the measurement results will be meaningless. In **Chapter 4**, results are obtained to evaluate the possibility of the multi-particle coincidence inside the PDA measurement volume, and the influence of the surface condition and morphology of the solid particles in the plasma spraying condition on the PDA measured particle diameter and volume flux. The possibility of the multi-particle coincidence occurred in the PDA measurement volume will be numerically evaluated based on the Poisson statistics equations. The waveforms of the Doppler burst recorded by the PDA signal processor for the water droplet, the plasma-sprayed NiCrAlY and YSZ particles will be compared to achieve a qualitative overview of the influence of the particle morphology on the PDA detected signals. In order to quantitatively evaluate the reliability and accuracy of the PDA measured particle diameter,

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the PDA diameter measurement will be performed for the NiCrAlY powders in different known size groups, each of which will be subjected to different arc power of the plasma jet so as to achieve different level of surface roughness and sphericity. Finally, the PDA measured particle volume flux of the YSZ powder will be compared with the volume of the actual deposit.

In **Chapter 5**, the radial distributions of the in-flight YSZ particle velocity, diameter, concentration and volume flux will be measured by the PDA under different settings of the plasma spraying parameters. Investigations will be performed for different powder injections schemes, for example, the internal injection of powders through one transversal injector and opposite two injectors, the external injection of powders through one injector and opposite two injectors for the injection angle of 90° and 60° counterflow direction. Corresponding to some typical settings for the PDA measurement, the coating/deposit will be produced for the microstructure analysis and deposition efficiency measurement in **Chapter 6**. The correlation between the PDA measured in-flight particle characteristics and the coating/deposit properties will be established on the purpose of finding a better approach to predict the coating quality through the in-flight particle information.

Finally in **Chapter 7**, all the results will be summarized and the overall conclusions will be provided. Suggestions and recommendations will also be proposed for further investigations.

Figure 1.1 Schematic illustration of a coating formation using plasma spraying with internal particle injection.

Figure1.2 Schematic of a typical cross section of a plasma sprayed coating [2].

Figure 1.3 Sectional view of the SG-100 plasma torch.

Chapter 2

Literature Review

2.1 Introduction

In the past few decades, numerous literatures have been published addressing many aspects of the plasma spraying process, which have greatly enhanced our understanding on the fundamentals and have aided to its development [3-7, 11-14]. In this chapter, the literature survey concentrates on the experimental investigations as well as on the theoretical analysis of the plasma spraying process. A systematical review of the diagnostic methods encountered most frequently in the three basic sub-processes of plasma spraying, i.e., plasma jet generation, plasma-particle interaction, and coating formation, will first be presented, followed by the important results and findings contributed to these processes. The review is not intended to exhaust all the papers published in this field. Emphasis has been put on the investigations of the plasma spraying using DC plasma torch.

2.2 Diagnostic Techniques for Characterizing the Plasma Spraying Process

2.2.1 Measurement Methods for the Plasma Jet

The plasma jet is the medium inside which the solid particles are heated and accelerated to form the final coatings. Understanding of the plasma flow field represents the starting point for studying the phenomena of particle heating and acceleration, which occur within the

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duration with the same order of magnitude as plasma fluctuation, at the same time providing the benchmark for calibrating and checking theoretical models.

The important parameters of the plasma jet are plasma temperature, velocity, entrained air fraction and concentrations of very reactive species such as atomic oxygen [4, 8, 13]. Flow field visualization techniques [26] using high-speed photography, Shadowgraphs, LaserStrobe and Schlieren systems have yielded qualitative information on the plasma jet behavior and turbulence as well as quantitative information on the plasma jet spreading and laminar-to-turbulent transition [27]. The temperature measurement techniques of the plasma jet are mainly based on Enthalpy probe, Emission spectroscopy, Thomson and Rayleigh laser scattering, and Coherent-Anti-Stokes-Raman Spectroscopy (CARS). The Enthalpy probe has been considered to be a reliable diagnostic tool for measuring plasma temperature from 2000 to 14000 K. When integrated with a mass spectrometer, it is able to obtain information on total entrained air fraction, and yield temperature and velocity information with the assumption of local thermodynamic equilibrium (LTE) [16]. Its main advantage is the simplicity of the technique and its possible use for the simultaneous measurement of multiple plasma parameters with a generally acceptable spatial resolution. The main limitation is that it is an intrusive technique, with the probe inevitably causing a local perturbation of the flow. Varieties of the non-intrusive optical methods have been applied in plasma diagnostics successfully. Emission spectroscopy used absolute intensity line techniques, Boltzman plots, Stark broadening, and two-point light emission correlation analysis to measure plasma temperature over 9000 K [16]. High-resolution coherent Thomson and Rayleigh laser scattering have provided a direct measurement of the plasma temperature, and very high-resolution measurement of the Doppler shift due to the bulk gas movement provided the plasma velocity information [28, 29]. Nonlinear spectroscopic

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techniques including Coherent-Anti-Stokes-Raman Spectroscopy (CARS) provided measurements of the concentration and temperature of molecular species like oxygen and nitrogen [30, 31]. The measurement of atomic species concentration was obtained by multiple-photon laser induced fluorescence (LIF) [31]. Unlike the large amount of literatures available for the plasma temperature measurement, the plasma velocity measurements have been relatively less studied. Laser Doppler Anemometry (LDA) was used to characterize the plasma velocity field by measuring the seeding fine particles ($<5\mu\text{m}$), assuming that seeding particles followed closely with gas movement [32]. Other techniques based on the laser light scattering by the plasma [33], have also been carried out. Recently, increasing attention has been given to obtaining the plasma velocity through a spectrum analysis of light and acoustic fluctuations of the plasma [34].

The plasma jet is subject to certain degree of instabilities, which would lead to fluctuations of plasma temperature and velocity. The amplitude of the plasma fluctuations, together with the spatial distribution of plasma temperature and velocity fields, varied with different plasma spraying parameters [3, 16-18, 32]. Various diagnostic methods were put on analyzing the plasma torch instability caused by (1) the complicated arc movement behavior between the cathode and anode, which results in continuous variations in the energy dissipation and in other parameters such as temperature, pressure and density in the plasma jet [17, 18, 35] and (2) the turbulent mixing of surrounding cold air into the plasma jet, which decreases the velocity and temperature of the plasma jet significantly [17, 32].

Arcs are actually the electrically conducting region between the cathode and anode [32]. The arc is initiated between the tip of the cathode and the water-cooled anode nozzle, as shown schematically in **Figure 2.1**. The cathode used in the plasma torch usually has a

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cone-shape tip, which confines the arc attachment on the cathode tightly without much freedom of movement. In addition, the cathode is located at the center of the torch and the forces exerted by the symmetrically passing gas flow on the cathode arc column balance each other, which also forces the shape of cathode arc column to remain unchanged. The major instability of the arc is found in the region where the arc attaches to the anode.

The anodic root position is determined by the complex balance between gas drag and Lorentz forces [17, 32]. For the straight injection of plasma gas, the arc anode attachment is perpendicular to the plasma gas flow and is exposed to a strong gas-dynamic drag force. The drag force in the downstream direction is caused by the collision of gas particles with the charged particles in the arc, which caused an axial motion of the electrically conducting region in the direction of the gas flow and consequently the elongation of the arc column. The boundary layer velocity profile with lower velocity (and therefore lower drag force) near the wall results in the curvature of the arc column in the attachment region. This curvature of the arc column produces a magnetic body force known as a Lorentz force. The drag force and the Lorentz force reach a dynamic balance for a stable arc root attachment position and deviations from that attachment position would result in an imbalance, which restores the attachment to its original location. The anode arc root might also travel circumferentially along the anode nozzle in the case of swirl plasma gas injection.

The complicated movements of anodic arc root attachment will lead to significant fluctuations in the plasma, which have been characterized by means of observing torch voltage fluctuations, acoustic emission, high-speed imaging, and plasma light intensity fluctuations [16-18, 29-34]. Monitoring the voltage waveform has been widely accepted as the most convenient and direct method for analyzing the arc instability because the most

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prominent consequence of arc movement was a strong fluctuation of torch voltage (in most cases the torch current was kept constant). End-on imaging using the high-speed video camera was effective in observing the cross section of the arc inside the nozzle and in estimating the thickness of the cold-gas boundary layer and the anode attachment location [35].

The fluctuating behavior of a plasma torch can be roughly characterized by three types of voltage waveform depending on the gas mixture and current [17, 18]. **Figure 2.2** shows the time dependence of torch voltage for the three different main operation modes.

1. Restrike mode — mode with the highest voltage fluctuation and asymmetric sawtooth shape of the arc voltage signal. The large voltage drops repeat periodically with a frequency between 3 and 4 kHz and have been attributed to “upstream restrikes”, for which a breakdown of the arc occurs with a new arc root closer to the cathode. The length of the new arc is strongly reduced, which results in a large drop of the voltage. Between these periodic features there are irregular drops of smaller amplitudes. These have been attributed to “downstream restrikes” for which a breakdown of the arc occurs with a new anodic root further from the cathode or on the diametrically opposite side of the cathode. For this phenomenon, which resembles reconnection, the attachment of the anodic root would be maintained at the same position and the arc would not be interrupted.
2. Takeover mode — prevails for the operation with noble gas. The voltage signal exhibits weakened fluctuations compared with those of the restrike mode. The name

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comes about from the fact that a new attachment is not created by breakdown but instead the new attachment gradually “takes over” the role of the old attachment.

3. Steady mode —operation at high current with a straight injection flow of pure argon, for which the voltage is kept nearly constant. In this mode the drag force of the plasma gas and the Lorentz force reach quasi-equilibrium. The anode attachment of the arc stays at one spot closest to the cathode, and the arc has an almost fixed length, which causes considerable erosion of the anode.

The plasma jet fluctuations associated with the arc root movement, reflected by the continuous temperature and velocity variations of the plasma, played an important role because their characteristic periods were of the same order of magnitude as the transit time of the particles in the plasma [17]. During the spraying process, this might lead to a non-uniform heating and transport of the injected powder particles and, consequently, adversely affected the quality and yield of the sprayed deposits. However, most of the plasma torches used for plasma spraying required a movement of the anodic arc root over the inner surface of the gun nozzle to distribute the large heat load and consequently, increased the anode lifetime [36]. The ultimate goal is to force the arc root to move in a controlled manner while maintaining a minimum arc fluctuation.

In addition to the fluctuation of the plasma jet originating from the complicated anodic arc root movement, turbulent mixing and entrainment of surrounding cold air into the plasma jet contributed significantly to the instability of the plasma jet [3, 17, 32]. When the plasma jet with extremely high velocity and temperature discharged into the stagnant atmosphere, the tremendous velocity and density differences between the plasma jet and the

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surrounding gas initiate their mutual turbulent mixing, pumping the ambient gas into the plasma jet. Large scale eddies extended from the outer edge of the shear layer towards the center of the jet where they were broken down into smaller eddies and mixed with the flow. This phenomenon was extensively investigated by Spores and Pfender [32] using the Laser Doppler Anemometry (LDA) through measurements of the velocities of very small alumina powder ($0.3\text{--}1\ \mu\text{m}$) seeded into the plasma jet and the surrounding air respectively. Based on the previous studies of cold circular jets, they described the mechanism of the large-scale flow structures which governed the entrainment processes in a circular plasma jet and how these structures broke down and led to the onset of turbulence. The schematic diagram of such an entrainment process is illustrated in **Figure 2.3**.

The entrainment of ambient air in to the plasma jet was also investigated and confirmed by shadowgraphs and CARS spectroscopy [37], by additional sampling experiments [38] and by probe sampling of the plasma jet [39]. This turbulent behavior enhanced the energy exchange between the plasma jet and the surrounding atmosphere, which accelerated the decay of plasma temperature and velocity with increasing distance from the nozzle exit. Entrainment of cold external air into the plasma jet usually produced undesirable coatings in plasma spraying. Spraying metal or alloys in atmospheric air made this situation undermined because of the oxidation of the metal components [2, 37].

2.2.2 Measurement Methods for the Plasma Sprayed Particle

Over the years, many optical-based techniques have been developed for monitoring the in-flight plasma-sprayed particles. These methods could be generally divided into point diagnostics versus imaging techniques and further, into methods utilizing external

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illumination versus passive methods [5, 7, 12, 13]. The latter methods do not use additional light sources to detect the particles, but rely purely on the spontaneous light emitted by the hot particles themselves. Point diagnostic methods require that only one particle be present inside a very small measurement volume during one period of measurement time. Based on a large number of samplings, the statistical information (mean, RMS, etc.) of local particle temperature, velocity, size and concentration can be derived. By traversing the small measurement volume over the whole field of interest, the spatial distributions of particle in-flight characteristics can be obtained and compared quantitatively under different spraying conditions. The strength of imaging techniques lies in its capability to provide a visual overview of the particle plume within an extended region of measurement by recording the thermal radiation from each particle whose surface temperature should exceed certain threshold to be detected. The information from the larger region of the spray jet gives a qualitatively direct indication of the trend of the hot particle trajectories and their dispersion (particle jet position and width) with different operational parameters.

Laser Doppler Anemometry (LDA) has been a well-established single point measurement technique since its first introduction in the early 1970s because of the very apparent reasons such as non-intrusiveness, directional sensitivity, high spatial and temporal resolution and high accuracy [32, 40]. Two coherent Gaussian laser beams intersect at one point to form the measurement volume, which exhibits a parallel bright-and-dark interference pattern. When a particle passes through the measurement volume, it scatters light and the intensity changes according to the interference fringes. Particle velocity is inferred from the frequency of the light scattered by a single particle, which is recorded in the form of Doppler burst by only one photodetector that is commonly integrated with the laser beams transmitter in the backward angle. LDA has been successfully applied in

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plasma spraying process for either plasma jet or in-flight particle velocity measurement. Particle size measurement has also been attempted by correlating the absolute intensity of the scattered light with particle diameter and particle concentration measurements by counting the number of particles per unit time [41]. However, both the particle size and concentration measurements were subjected to large inaccuracies due to the dependence of the scattered light intensity on the particle trajectory within the measurement volume, whose light intensity follows a Gaussian distribution. Affected by the background radiation from bursts of plasma and clouds of fume originating from evaporating powder material, the measurements were further deteriorated when applying the LDA to the plasma spraying process for particle diameter and concentration measurement [42].

Durst and Zare [43] first published the idea of measuring particle size using phase measurements by the LDA. Later, many research groups such as Bachalo and Houser [44], Saffman et al. [45], Bauckhage et al. [46], Qiu and Sommefeld [47], and Naqwi and Durst [48], contributed to the invention and improvement of the phase Doppler technique. Companies such as Aerometrics, Dantec and Invent have also explored various aspects of the technique and introduced commercial products. After almost two decades of development and evolution, the Phase Doppler Anemometry (PDA) has become more mature with the potential error sources being identified and innovations being provided to overcome these problems [49, 50]. An extension from the LDA technique, the PDA shares almost the identical optical arrangement of the LDA except that the PDA employs more than one photodetector, which are located at an off-axis angle from the plane formed by the two laser beams. All the signals detected by each photodetector when a single particle passes through the measurement volume are periodic in time and exhibit the same frequency, which is proportional to the particle velocity. The phase shift between a pair of

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signals is related to the diameter of the spherical particle. Under certain conditions, the phase-diameter relationship is almost linear. Hence, it allows simultaneous determination of the velocity and size of spherical solid particles, droplets or bubbles, making it possible for the local particle size distributions and size-velocity correlations to be obtained. Moreover, recent developments have provided the basis with more accurate particle concentration or volume flux measurements and even for the estimation of the refractive index of particle materials in order to distinguish particles with different optical properties. Although the PDA instrument has become a standard diagnostic technique for spraying characterization of the size and velocity of drops and other spherical particles in a wide range of applications, its application in characterizing of the plasma sprayed particles was only available in recent years [51-53]. The detailed description of the PDA measurement principle will be given in **Chapter 3** and the specific considerations for applying the PDA in the plasma spraying process will be addressed in **Chapter 4**.

Measurement of the plasma-sprayed particle surface temperature in the current stage is commonly based on the technique of two-color pyrometry [54, 55], which involves the formation of an image of the emitting particles when they pass through the measurement volume defined by the collection optics. The collected radiation is composed of three components: the thermal radiation emitted by the particles under observation, the light emitted by the plasma contained in the measurement volume and directly collected by the detection optics, and the light scattered by the particles irradiated by the arc and the surrounding emitting gases. Two-colour pyrometer as a device for optical temperature measurement is based on the measurement of the light emitted by the object in two separate wavelength ranges with centre wavelengths. **Figure 2.4** shows the light intensity in relative units for an object made of tungsten in different temperatures. It can be seen that

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when the temperature rises, the total intensity rises rapidly. In addition, the radiation is shifted into shorter wavelengths. Two-colour pyrometer eliminated the effects of particle size, emissivity (assumed the particle is a gray body, i.e. the emissivity does not depend on the wavelength) and non-ideal focusing of the optics on temperature measurement. The most important systematic error source is the non-grayness, i.e. the wavelength dependency of the emissivity of the particle material. It can be considerable especially for metallic materials. Moreover, it has been demonstrated that the plasma light scattered by the particles led to an overestimation of the actual particle surface temperature [56].

DPV2000 (Tecnar Automation, Canada) is a very popular device which measured the individual particle surface temperature based on two-color pyrometry and particle velocity by the time-of-flight method [57, 58]. Particle characteristics are monitored by detecting the thermal radiation emitted by individual particles crossing the field of view of a two-slit optical mask located on the tip of an optical fiber. When a particle passes through the measurement volume, it is seen twice through the two-slit mask. This event gives rise to a two-peak signal at the output of the photodetectors located in the detection box. The particle velocity is computed from the time of flight of the particle in the field of view of the two-slit mask. The particle temperature is obtained from the ratio of the thermal radiation detected at two different wavelengths (two-color pyrometry) and the particle diameter is calculated from the absolute intensity of the thermal radiation at one single wavelength after measuring the temperature. The sensor head is comprised of the two-slit mask at the tip of a single optical fiber and a linear array of optical fibers that transmits the thermal radiation emitted by the particle jet to a linear CCD camera. The sensor head can be attached to the torch or located in a fixed position in the spray booth, making it possible to check the state of the spraying process.

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Another commercial system, the IPP-2000, measures the temperature of the particle jet as a whole [59, 60]. The sensor head contains the end of an optical fiber and a lens. The thermal radiation emitted by the particle jet is sent to the detection box that contains two photodetectors sensitive to two different wavelength ranges. The temperature of the particle ensemble is determined from the ratio of the detector outputs (two-color pyrometry). Contrary to the previous system, the IPP-2000 does not give any information about the velocity and diameter of the sprayed particles. It is, however, eventually easier to use since the volume of measurement is larger, making the alignment simpler.

The approaches mentioned above are very effective in measuring particle characteristics in a laboratory environment but their use in industry for controlling spraying process is difficult owing to their complexity, fragility, high technical skill and safety requirements. Imaging systems to measure particle trajectory and velocity also have the same type of limitations for use in industry. Recently, different techniques were developed which were aimed at or adapted to monitoring particles in a production environment. These techniques, in general, took advantage of the thermal radiation emitted by the hot particles to monitor some of their characteristics without using intense laser beams. For example, a linear camera (coupled or not with an optical fiber bundle) can be used to monitor the orientation, width and intensity of the particle plume [61]. The field of view of the sensor is oriented perpendicularly to the particle spray direction. With such an arrangement, one can rapidly characterize and optimize the particle injection conditions. The same arrangement can also be used to detect unstable spraying conditions during coating production. The linear camera is quite easy to be used and relatively inexpensive. It can supply reliable information about the powder injection conditions by monitoring the particle trajectories and their dispersion (particle plume position and width) but it gives no direct information

about the particle temperature and velocity. In recent years, a commercial product utilizing the CCD camera named SprayWatch has drawn great attraction for its ability to provide an overall image of the particle plume while at the same time, providing quantitative information of particle temperature and velocity [62-64]. This device is relatively easy to be used and no calibration process is necessary.

It is understandable that the above-mentioned techniques cannot exhaust all the methods used to on-line characterize the plasma-sprayed particles. However, they must be the most representative and widely used techniques in this field. Due to the development of these techniques, the momentum and heat transfer between plasma and particles are understood better now than before.

2.2.3 Measurement Methods for the Plasma Sprayed Coating

The general procedure for investigating individual particle flattening and splat formation process is as following [65, 66]. A shielding plate with a small hole is placed in front of the plasma gun and only particles passing through the hole can reach the substrate. The substrate is pre-heated by electric heater or directly by the plasma jet, and regulated by the temperature controller or cooling air. Substrate temperature is measured by a thermocouple fixed on it and the oxide layer formed after heating the substrate is measured using an X-ray photoelectron spectrometer (XPS). Once sprayed particles are seen passing through the hole in the shielding plate, the substrate was rapidly passed through the stream of particles to collect only a few splats. Particle parameters at impact can be measured using various optical devices as described in **Section 2.2.1**. The flattening of particles on the substrate can be studied using either a linescan test or fast two-color pyrometer. Each splat corresponding to these measurements will be characterized subsequently by atomic force

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microscopy (AFM), scanning electron microscopy (SEM), or transmission electron microscopy (TEM).

The lifetime and thermal-mechanical properties of the plasma sprayed coatings are closely related to their microstructures, which are characterized by large numbers of porosities and cracks of various sizes and lengths [2, 6, 21, 67, 68]. Porosity is an integral part of the plasma sprayed coatings, which was commonly characterized by mercury intrusion porosimetry (MIP) [69], image analysis (IA) with the aid of scanning electron microscopy (SEM) or optical microcopies [21, 67, 68], some non-destructive testing (NDT) methods utilizing ultrasonic or capacitance techniques based on the known thickness of coatings [70], or combinations of them. Density measurements can also be used to reveal a measurable difference in the percentage of porosity [71].

Another important property of the plasma sprayed coatings is the adhesion/cohesion strength, which is usually measured by the use of tensile tests in which the load is applied through an epoxy adhesive bond to the outer surface, or by the fracture mechanics methods which allow separate determination of cohesive and adhesive fracture parameters [72].

As introduced by Planche et al. [73], a macrohardness tester was used to generate cracks in the coatings (cross sections) in order to evaluate the coating toughness. A WOLPERT macrohardness tester was used with a 2kg load (optimized for the coating by Planche et al. [73]). Crack lengths could be examined with an optical microscope. It was found that the adhesion between the lamellae, which characterized most of the coating cohesion, appeared mostly at the lower torch energies.

Deposition efficiency (DE) indicates the percentage of particles that adhere on the substrate to form splats versus all the particles injected inside the plasma jet. DE was commonly calculated by the ratio of the deposit weight gain to the total weight of the particles being sprayed [67, 74, 75].

2.3 Plasma Jet Characteristics

2.3.1 Plasma Jet Generation

Plasma jet is produced by ionizing the plasma gases in the electric arc between the negatively charged cathode and positively charged anode inside a plasma torch, which induce a high energy state of plasma with very high temperature and velocity. Both electrodes are separated by an insulation insert. Pressurized water which cools the torch enters in the cathode part and leaves the anode part circulating through the insulation tubes or channels in the insulation insert. Torch design must ensure perfect centering of the cathode against the anode. Plasma gas can be supplied to the cathode zone either axially, through the openings in the insulation insert, or tangentially, in the form of a swirl. The nozzle, serving as anode and subjected to considerable heating effect, would transfer several Kw of output power per square centimeter. The nozzle is manufactured from high-purity copper with high thermal conductivity and in most cases it is directly water-cooled. The cathodes are usually made of tungsten enriched with 2% ThO₂. Thorium in tungsten serves predominantly in the lowering of electron emission potential and hindering of cathode wears due to impurities in the plasma gases.

In order to design more efficient and durable torches in the future, it is important for designers of plasma torches to obtain a better overall understanding of the various

interdependent parameters that govern the operation of torches [14, 76]. In the past few decades, extensive efforts, both experimentally and numerically, have been made aiming at investigating the influence of torch parameters on the plasma jet properties, primarily the velocity and temperature fields.

2.3.2 Experimental Work

2.3.2.1 Influence of Arc Current

Most of the plasma spraying systems have a fixed arc voltage setting with an adjustable arc current value. The arc current level has the most significant influence on the plasma jet properties [2, 25, 29]. An increase in the arc current would result in an increase of plasma energy density, which raises the plasma jet velocity and temperature correspondingly. However, the surface temperature of the injected particles might decrease when arc current is too high due to the higher particle velocity and their shorter residence time inside the plasma jet. The influence of the arc current on the isotherms length and width of the plasma jet has been studied for arc currents below 450 A [32]. The results have shown a higher surrounding air pumping effect with increasing arc current. However, when the arc current is raised to higher values, a limit is reached such that the length and width of the plasma jet isotherms would not change prominently. Also, in this condition the further increase in arc current would not modify the surrounding air entrainment on a significant scale.

2.3.2.2 Influence of Plasma Gas Composition, Injection Method and Flow Rate

Plasma forming gases are commonly argon or a mixture of argon with another noble gas such as helium or molecular gas such as hydrogen and nitrogen. Selection of gas combination and proportion is based on the desired temperature and velocity of the plasma jet and the degree of inertness in the sprayed material and substrate. Pure argon has a low ionization potential and enables easy arc ignition [1]. Since argon easily attains high temperature, the arc voltage can be maintained at a low level. In contrast, nitrogen and hydrogen, as two-atom gases, are harder to ionize because they must be dissociated first. Therefore their arc and ignition voltages are considerably higher than those of argon. Addition of helium and molecular gases results in a drastic increase of the plasmas enthalpy, which leads to substantial increase of the arc voltage (for a given current) and the power input to the torch.

Figure 2.5-2.7 shows the temperature dependence of plasma viscosity, thermal conductivity and electric conductivity of Ar-He and Ar-H₂ mixtures at atmospheric pressure [1]. It was illustrated that in the range of plasma temperature (10,000 to 14,000K) for particle heating and acceleration, the viscosity of mixtures containing helium was increased drastically while addition of hydrogen decreased the plasma viscosity significantly. This conclusion was confirmed by Roumilhac et al. [77] and was used to explain the discrepancy that particles obtained higher velocity in the Ar-He plasma jet, whose velocity was lower than that of Ar-H₂ mixture, giving that the plasma jet formed by these two types of gas mixture had the similar isotherm and heat treatment of particles. The higher viscosity of Ar-He mixture was responsible for increasing the particle acceleration and it compensated the lower velocity of the Ar-He jets. Thermal conductivity and electric conductivity of the plasma forming gas had a direct influence on the actual power

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dissipation in the arc column. With the same nozzle diameter, arc current and plasma gas flow rate, power dissipation increased with higher thermal conductivity and lower electric conductivity of the plasma gas. This explained why the plasma jet formed by gas mixtures containing hydrogen possessed higher temperature and caused better particle melting. Taking advantage of the higher viscosity of helium and higher thermal conductivity of hydrogen, the plasma jet formed by the gas mixture of Ar-H₂-He in the optimal volume composition was proven to be able to provide better heat and momentum transfer to injected particles and lower the entrainment of the surrounding air [78, 79].

Both the nature of plasma gas mixture and the gas injection method have a direct influence on the mode of arc instability [17, 24]. Restrike mode is popular under the condition of low current, high hydrogen percentage and straight gas injection. Most plasma spraying operations with argon-helium mixtures, either in straight or swirl gas injection, fell into the range in which the takeover mode dominated. With a very high current, pure argon gas and straight gas injection, the arc assumes the steady mode, which is represented by a very low mean voltage and little fluctuation. The steady mode of arc is seldom observed with the swirl gas injection [17].

Increasing of the plasma gas flow rate resulted in an increase of plasma jet velocity but decrease in the plasma temperature [3-5, 33]. Moreover, higher plasma gas flow rate enhanced the turbulent mixing of plasma jet with surrounding air. The mixing of cold air into the plasma jet further reduced the temperature of the plasma significantly because air had a higher specific heat than argon, resulting in lower temperatures for the same energy content [32].

2.3.2.3 Influence of Surrounding Atmosphere and Chamber Pressure

In Air Plasma spraying (APS), the turbulent mixing of the plasma jet with cold ambient air changes the plasma jet chemical composition and reduces its temperature and velocity, which adversely affects the coatings formed [80]. Demands for coatings of high quality and chemical purity have led to the development of plasma spraying conducted in controlled atmosphere. The name, Controlled Atmosphere Plasma Spraying (CAPS), is a generic category including Inert Plasma spraying (IPS), Vacuum Plasma spraying (VPS), Low Pressure Plasma spraying (LPPS), and High Pressure Plasma spraying (HPPS).

Controlled Atmosphere Plasma Spraying (CAPS) yielded a coating with unique properties that would not be possible to be achieved in standard atmospheric environments [80-83]. Coatings were very dense, extremely well bonded and metallic coatings were free of oxides and other contamination. Non-metallic coatings, such as ceramics sprayed in chambers backfilled with non-reactive atmospheres, were very pure.

CAPS systems were very flexible in the types of atmospheres that could be used (Argon, Nitrogen, etc.) [81] and the internal chamber pressures that could be selected for spraying conditions, which could be in the range from near vacuum (as low as 50 mbar) to elevated pressures (as high as 4 bar) [82, 83]. Inert, protective atmospheres were used for ensuring the purity of the reactive sprayed materials or protecting workpiece substrates that would be oxidized or contaminated easily. Comparing between the plasma jet flowing in air and argon atmosphere, it was found that with almost the same central part of the plasma core temperature distribution, the plasma jet expanded significantly for argon surrounding. This phenomenon was explained by the fact that the dissociation of the air molecules happened at relatively low temperature levels compared with the ionization temperature of argon.

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The dissociation process, with high energy consumption, cooled the plasma jet efficiently. Particle velocity had no significant difference for argon and air atmosphere; however the particle surface temperature was higher in argon. To reduce the coating production cost by 25 to 30 percent, a nitrogen atmosphere was used as a substitute for the conventional argon atmosphere. It was found that the coatings produced in the nitrogen atmosphere had similar properties as those produced in the argon atmosphere.

The plasma jet is strongly affected by the chamber pressure around the plasma torch (**Figure 2.8**). As the chamber pressure decreases, the plasma jet extends and the plasma gas velocity continually increases. Such plasma jets in the LPPS and VPS processes are especially suitable for preparing dense metal coatings such as bond coats for thermal barrier application and have had a major impact on coatings for aircraft engines. However, experiments showed substantially lower plasma temperatures and densities at low chamber pressures and the heat transfer from plasma jet to the particles was drastically reduced in spite of much longer isotherms. When the chamber pressure was lower than 6.7 kPa, it would be difficult to achieve good melting of refractory materials such as ceramics [82, 83].

On the other hand, when the chamber pressure is elevated, the high-pressure atmosphere around a torch condenses the plasma flame and enhances the heat transfer from the plasma gas to the injected particles [80]. Plasma temperature rise accompanied with the plasma flame shrinkage under high pressure has brought about effective heating of particles, resulting in high deposition efficiency and densified coating with improved hardness, in spite of reduced particle velocity. There are of course potential drawbacks to spray at higher pressure, such as the possible gas entrapment resulting in increased coating porosity.

Moreover, the condensed plasma heats the torch itself so severely that the torch components, such as the nozzle, often melt down. Therefore, determination of the effective and safe parameters is the most important subject in order to establish the HPPS as a practical coating tool.

2.3.2.4 Influence of Nozzle Design

The main goal for nozzle design is to extend the hot core of the plasma jet in order to improve the melting and acceleration of the injected powder and to diminish the influence of entrainment of surrounding air [14]. For vacuum plasma spraying (VPS), it was well known that at pressure conditions where supersonic plasma jets were generated, a converging-diverging (Laval) nozzle contour improved the jet quality by increasing velocity and extended jet core compared to the standard cylindrical anodes [84]. Laval anode nozzles were also of advantage for atmospheric plasma spraying (APS) and subsonic velocities of the plasma jets by diminishing the influence of cold gas entrainment and generating a plasma jet with higher centerline velocities and larger high temperature zones. The investigations have shown that it was not only the internal diverging contour but also the external anode respectively torch contour that had influence on the interaction of plasma jets and their environment. When nozzle diameter was increased, the plasma jet became larger and slower. This velocity drop led to a decrease of the surrounding air entrainment, but also reduced the particle velocity.

2.3.2.5 Influence of Electrode Condition

Nozzle wear is very critical not only to the torch lifetime but also more important to the coating quality. This is because the nozzle wear changes surface profile of the nozzle, as demonstrated in **Figure 2.9**, which affects the plasma formation. Due to the high temperature and velocity gradient, a small change in the plasma shape might have a significant effect on the particle trajectories and variations of the coating properties [76, 85].

The lifetime of electrodes is strongly affected by the plasma gas composition and it was well known that higher hydrogen content led to a quicker erosion of the electrodes [77]. In the case of Ar-H₂ mixture, higher arc constriction would be expected, which led to a smaller emissive area and a higher wear of the cathode. The electrodes wear affected the plasma jet geometry significantly. The new anode could provide a longer and more stable plasma jet compared to the old ones. The shorter plasma jet length might be induced by the decrease in the output power with an older anode. However, the power drop could be compensated by increasing the current or hydrogen flow rate. Plasma jet stability was affected by the arc attachment movement, which was also a function of the anode surface.

2.3.3 Numerical Modeling

The numerical model developed by McKelliget et al. [86] represented the pioneering work, which was applied in the form of two-dimensional elliptic partial differential equations in conjunction with $k-\varepsilon$ turbulence model and energy transport equations to predict the velocity and temperature fields of the plasma jet. The theoretical predictions were then compared with the experimental investigations. Good agreement was obtained for velocity

distribution, but for the temperature distribution, the comparisons were not appreciable due to some oversimplified assumptions.

Westhoff and Szekely [87] presented a mathematical model describing the electromagnetic phenomenon within a nontransferred arc plasma torch and the resultant plume. The important feature of this model was that it could predict both the pattern of heat generation within the torch and the electromagnetic forces, and thus provided a fundamental basis of predicting plume behavior. In the development of the model, allowances were made for the conservation of mass, momentum, thermal energy and the continuity of current.

Previous models neglected the involvement of chemical reactions between species. Ramshaw and Chang [88] developed a computational model, the LAVA code in order to represent the plasma as a multicomponent chemically reacting ideal gas with temperature-dependent thermodynamic and transport properties. The LAVA code was used to simulate a steady-state axisymmetric argon plasma jet flowing into cold argon and comparison was made with the experimental data. From the results, the authors suggested that the uncertainties about the inlet flow profiles should be reviewed to reduce the deviation occurred.

Chang and Ramshaw [89] also performed simulation of a steady-state axisymmetric argon plasma jet flowing into cold air by using LAVA code and comparisons were made with experiment data. The overall agreement was fairly good but yet not fully satisfactory because of too much diffusion at the upstream and too little at the downstream. The deviations were claimed by the authors as due to inadequacies of turbulent model, uncertainties in the inlet flow profiles of velocity, temperature, swirl and turbulent

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parameters, thermal and chemical non-LTE effects, numerical discretization, no interaction between turbulence and the ionization and dissociation chemistry.

The LAVA code had been modified by Fincke et al. [90] to simulate Ar-He plasma jet expanding in the cold air. This modified code had the capability to simulate demixing of Ar and He in the downstream region of the nozzle exit and remixing at the far downstream region while the initial LAVA code did not address. However, experimental value dependent boundary conditions were still needed to be imposed. This resulted the variable performance of the LAVA code.

Since there was still no well-established code for chemical reaction, Bauchire et al. [91] developed a 2D mathematical model, considering only heat, fluid as well as magnetic hydrodynamic flow to describe the effect of turbulence toward temperature and velocity profile of the plasma flame. Two different types of Prandtl's mixing length models and k - ε model were used. The effects were insignificant. However, the authors proposed the usage of k - ε model since more information on turbulence were provided. The detailed information might be useful for other purposes, such as the local values of turbulence intensity or kinetic energy was necessary for determining turbulence dispersion of particle.

Liu et al. [92] attempted to avoid the application of complex chemical reaction model and simulated a simplified model, which is based on energy, mass conservation and turbulence model with temperature dependent thermodynamic and transport properties, to gain the temperature, velocity and air content of the plasma jet. Then, comparison was made between the authors' results with those of Eichert et al. [93] who used a much more complicated chemical reaction. Results showed there was no significant difference.

2.4 Heat and Momentum Transfer from Plasma to Particles

The plasma-particle interaction process represents the intermediate state which connects the plasma jet formation to the coating built-up. The consequence of this interaction is the heat and momentum transfer from the plasma to particles. Particles are injected into the plasma jet to be heated and accelerated. Their velocities and surface temperature are determined by the drag force and temperature histories seen by the particles along their trajectories inside the plasma jet. Ideally, the majority of the powders should penetrate inside the hottest zone of the plasma jet and have the longest residence time there. For a given particle, its trajectory inside the plasma jet depends on the conjunct effect of the plasma parameters and powder injection parameters [20]. As reviewed systematically in the previous section, plasma parameters determine the plasma jet properties, any change of which will inevitably alter the characteristics of the in-flight particles and the resulting coating. Keeping the plasma parameters constant, the particle injection parameters control to a large extent the particle trajectory and dispersion. To obtain desired coatings with specific mechanical and physical properties, the influence of both the plasma parameters and particle injection parameters must be identified.

2.4.1 Experimental Work

The traditional way of assessing the influence of the plasma spraying parameters to the resulting coating was to change them one by one and examine the resulting properties of the coating such as the coating density, porosity, cohesion/adhesion and deposition efficiency. This complicated and time-consuming evaluation method required surveillance and control over so many spraying parameters. Moreover, some parameters, such as electrode wear and fluctuations in plasma jet and powder injection conditions were

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uncontrollable. Due to the high velocity and temperature gradients in the plume, even small changes in the controllable or uncontrollable parameters could result in significant variation to the in-flight particle behavior and consequently to the microstructure of the coatings [12, 13]. The alternative was to use non-intrusive optical-based diagnostic devices to monitor the particle in-flight characteristics, principally particle surface temperature, velocity and concentration, from which to predict the coating properties. Thus, rather than controlling the operation conditions of the torch, manipulating of the sprayed particle state could lead to a better control of the spraying process.

As mentioned above, the direct parameters controlling the coating microstructure and thermal/mechanical properties were the molten state and velocities of the impacting particles. Most of the investigations utilizing the optical diagnostic tools were aimed at providing either the spatial distribution of particle velocity, size and temperature inside the plasma jet or the variations of these parameters under different operation conditions [7, 13, 14]. It was well known that for a particle with given properties (chemical composition, size and morphology), its velocity and temperature upon impact were determined by, on one hand, the plasma parameters which controlled the formation and properties of plasma jet as mentioned in **Section 2.3**, and on the other hand by each particle trajectory in the plasma jet [19, 20]. The particle trajectory was controlled by the particle injection parameters, which might include particle injection velocity, the position and tilting angle of powder injector, injector internal diameter, powder feed rate and so on. Other than the number of parameters involved, the fact that the injected powders had size and injection velocity distributions that resulted in a trajectory distribution added to the complexity. Thus, it could be expected that the injected particles would have large differences in momentum

and temperature histories and the coating was actually resulted from the piling up of splats with very different properties.

2.4.1.1 Influence of Powder Properties

Selection of powders suitable for the specific plasma spraying condition was the first consideration. It was recommended that the powders should exhibit good pouring properties; also, the powder particles should be of spheroid or similar shape and their size should be as uniform as possible. The spraying of powder with particles of different sizes might cause overheating and eventually the evaporation of finer fractions while insufficient heating of larger particles [2]. Due to their lower inertia and thermal capacity, smaller particles experience higher acceleration and faster heating. However, they also decelerated and cooled down faster than larger particles.

Particle morphology also played an important role on the pneumatic feeding and coating properties. The powder mass flow rate depended directly on its apparent density, which was higher with dense spherical particles such as those made by the RF plasmas. Vardelle et al. [20] investigated the in-flight particle velocity and surface temperature for dense fused and crushed particles and porous agglomerated particles. It was found that the agglomerated particles partially exploded upon penetration into the plasma jet. This phenomenon was attributed to the poor mechanical resistance of the agglomerated particles and the high thermal shock experienced by the gas trapped in the pore structure, which made the agglomerated particles expand and explode upon exposure to the plasma jet. The exploded particles did not penetrate into the jet because of their low momentum. Thus, they traveled along the jet fringes, which resulted in a much lower velocity. On the other hand, the agglomerated particles that did not explode inside the plasma jet reached higher

velocities than the fused and crushed particles due to their lower density, but they decelerated faster. When considering the surface temperature, it was higher for fused and crushed particles than for agglomerated particles, which was logical because the velocity of the fused and crushed particles was lower.

2.4.1.2 Influence of Plasma Parameters

Plasma parameters had the direct influence on the properties of plasma jet as mentioned earlier, which were the heating source of the in-flight particles. Generally speaking, particle heating depended on the plasma enthalpy, thermal conductivity of the plasma and the residence time of the particles inside the hottest zone of plasma jet. Increasing of power level resulted in an enthalpy increase but at the same time a shorter residence time of particles due to the increased particle velocity [20]. Both the plasma enthalpy and thermal conductivity were enhanced by the hydrogen and helium percentage in the plasma. However, particle surface temperature was higher in the Ar-H₂ mixture for the reason that the mean thermal conductivity of this mixture was the highest. In addition, the viscosity of mixtures containing helium was increased drastically while addition of hydrogen decreased the plasma viscosity significantly. Since the higher viscosity of the plasma was responsible for the increase in particle acceleration, particles obtain higher velocity in the Ar-He mixture than in the Ar-H₂ mixture. Thus, particles had a longer residence time in the Ar-H₂ mixture, which further contributed to a better heating of particles. Keeping other parameters constant, net increase of the total plasma gas flow rate led to an increase of particle velocity but a decrease of particle surface temperature due to the shorter residence time. Increasing the nozzle diameter led to a decrease of particle velocity and an increase in particle surface temperature. A controlled atmosphere of argon broadened and

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lengthened the plasma jet, which resulted in broader velocity and surface temperature distributions of the particles, with similar maximum velocities but higher temperatures [82].

It should be reminded that the influence of plasma parameters on the particle velocity and temperature mentioned above was only the general trend without taking into account of the arc fluctuation and the plasma instability caused by the entrainment of surrounding air, the amplitude of which varied with plasma parameters. The arc fluctuation caused a periodic variation of the plasma jet, which resulted in non-consistent treatment of in-flight particles. It was also well known that the air pumping effect rapidly cooled the plasma jet with oxygen dissociated at 3500 K. Since pumping increased with the plasma gas velocity, all parameters increasing plasma gas velocity had a negative effect on particle heating. This happened when the arc current, plasma gas flow rate, or percentage of hydrogen increased, or when the nozzle diameter decreased. When the viscosity of the plasma increased, for example with the increase of He percentage, the pumping effect was reduced. The arc instability and turbulent effect of the plasma jet usually had an adverse effect on the in-flight particle heating and acceleration, which added to the complexity when considering the influence of plasma parameters. In conclusion, plasma parameters had the inter-related effects on particle in-flight behaviors and sometimes compromises had to be found [30].

2.4.1.3 Influence of Particle Injection Parameters

For a given plasma jet, i.e. constant plasma parameters, particle injection parameters controlled to a large extent the dispersion of particle trajectories. In most plasma spraying processes with a DC plasma torch, particles were suspended in the carrier gas flow and introduced into the plasma jet through one transversal injection port. The carrier gas flow rate had to be adjusted according to the specific powders used so that the maximum

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number of particles would penetrate into the hottest zone of plasma jet and experience the optimum acceleration and heating. If the carrier gas flow rate was too low, the particles could not penetrate inside the plasma jet; and if it was too high, they crossed it without undergoing efficient heating and acceleration. The optimum trajectory was that corresponding to the highest surface temperature and resulting in the highest deposition efficiency. Powder injector geometry also played an important role in the particle injection velocity since the particle velocity at the injector exit was strongly influenced by a particle's last few collisions with the inner wall of the powder injector. Fincke et al. [19] investigated the injection velocities of powder injected through some most commonly encountered geometries of the powder injector. It was found that curved geometries that caused the particles to decelerate at an abrupt corner upon injection into the plasma jet always resulted in the lower injection velocities while the straight geometry led to higher injection velocities. With the same carrier gas flow rate and injector geometry, increasing the powder feed rate slightly decreased the particle injection velocity and the surface temperature of in-flight particles [10, 19, 20].

Different powder injection methods have been proposed in order to increase the powder residence time in the plasma jet. In addition to the prevailing one-port transverse orthogonal injection, injectors designed to tilt counter to the flow for certain angles have also been investigated. In spite of the higher particle velocity and surface temperature produced, this type of injection was not preferable. Problems occurred when particles adhered and began to build up to form a deposit on the anode throat. It was believed that axial injection of powder would ensure that the particles experience a much more uniform heating in the plasma jet. However, it was also difficult to prevent the effect of particle deposition onto the electrodes.

2.4.1.4 Reaction of Particles with Surrounding Environment

During the particle flight in plasma, interactions of particles with the gases of surrounding atmosphere and the plasma took place, since all materials at or close to melting temperature exhibited high chemical activity. The entrained surrounding gas could react with the hot particles or the vapor originating from particles. Vaporization could be easily achieved with DC plasma jet either with relatively low-boiling temperature materials or refractory materials with low thermal conductivity, where the heat propagation phenomenon would dramatically increase the particle surface temperature. When spraying in air, particle oxidation took place in flight [2]. As mentioned earlier, the quantity of air entrained in the plasma jet depended strongly on its velocity and viscosity.

2.4.2 Numerical Modeling

Basically, Newton second Law of motion is used for momentum transfer and energy balance was applied for heat transfer. However, to make it more realistic, many researchers had taken other phenomena into consideration. Joshi and Sivakumar [94] had included the effects of steep temperature gradients, internal heat conduction inside the particle and temperature-dependent plasma properties and Knudsen effect simultaneously. They found that the Knudsen effect on both heat and momentum transfer became progressively more significant as the particle size decreased.

A comprehensive article on the particle momentum and heat transfer was published by Boulos et al. [95] In this work, an exhaustive description of issues related to plasma-particle momentum and heat transfer and other aspects of plasma-particle interaction was compiled. In particular, the effects of particle carrier gas flow rate on the plasma velocity

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and temperature, the effects of steep temperature gradient between plasma and particle, and the effect of the internal heat transfer were discussed in this article.

In a theoretical analysis carried out by Bourdin et al. [96], concentrations were put on issues such as the steep temperature gradient existing between the plasma and the particles, boundary layer relaxation time, internal heat transfer in the particles, and the effect of radiation heat transfer between plasma and particles. It was found that the radiative heat loss from the particles was negligible compared to the convective heat flux.

The combination of the two aspects, i.e. plasma flow modeling and plasma-particle interaction in the modeling of plasma spraying was reported by Lee et al. [97]. In their simulation, thermal plasma jet flow and multi-particle injection were considered. In the multi-particle injection model suggested by them, a coupling effect between the continuous phase and dispersed phase has been described.

Wan et al. [98] applied LAVA code and included a model associated with the sprayed particle in order to provide clear view on phase change, for instance melting, vaporization and resolidification. The particle geometry and heating model were as indicated in **Figure 2.19**. Since the material used herein (zirconia) had low thermal conductivity and large Biot number, this model coupled the temperature variation inside the particle (internal conduction) with the effect of heat transfer by convection and radiation, steep variation of density in the plasma plume, effects of temperature-dependent plasma properties and Knudsen non-continuum.

Moreover, Ahmed and Bergman [99] performed the simulation by decoupling the plasma and particle momentum and heat interaction. In the plasma flow, consideration of the existence of the substrate at 97 mm away from the jet exit was taken into account. While, the phase-change phenomena and the effects of turbulence in the jet on the particle motion and heating were included in plasma particle interaction. From the results presented, dissimilarity of particles sizes and density caused the difference in particles trajectories, leading to the difference lengths time of exposure in the plasma plume. Smaller particles encountered smaller drag force, had less momentum and subjected to more turbulence compared to the larger particles. As for particle heating, 30 μm particles melted early and remained in the molten stage along the rest of the flight, whereas, particles of 70 μm never melted. As for the 50 μm particles, they were in partial molten stage and suffered from the resolidification phenomena.

Williamson et al. [100] developed a numerical model to examine the effects of turbulence, injection direction and particle properties on particle behavior during thermal plasma spraying. Introduction of “one phenomenon at a time” was practiced in order to distinguish and characterize those effects and then all phenomena were considered simultaneously. From this numerical simulation, the authors reached a few interesting conclusions. Regarding particle dispersion in the injection direction, particle size distribution, injection velocity distribution, turbulence, injection direction distribution or particle density distribution (in order of decreasing importance) were significant effects. While, distribution of injection directions and turbulence played important roles in dispersion in normal-to-injection direction and particle size was main effect for determining particle velocity and temperature.

2.5 Plasma sprayed Coating Characteristics

Particles injected into the plasma jet were rapidly heated and accelerated, heading towards a substrate as a plume of molten or semi-molten droplets, which then flattened and solidified successively upon impacting on the substrate or previously deposited layers. Solidification of each particle was believed to be complete before the next particle hit at the same location due to the extremely rapid cooling of particles at impact. The spreading kinetics of each individual particles impinging on the substrate surface determined the mechanism of the formation of splats, which were the building elements of any given coating. Coating properties, such as the microstructure, adhesion/cohesion strength, and thermal-mechanical properties, were closely related to the individual splat flattening, splat-splat and splat-substrate interaction [6]. It was previously thought that the particle velocity, temperature, diameter and hitting angle at impact were the most important factors controlling the degree of flattening and solidification of the splats [67]. In most of the research carried out, the properties of the individual splats such as cooling rate and flattening degree were most frequently related directly to the particle parameters at impact produced under particular processing conditions with the aid of various on-line diagnostic methods. However, recent investigations accentuated the important role the substrate conditions such as substrate material, temperature, surface oxidation and roughness played in the splat formation [101, 102]. In spite of the great progress made in the past few decades, studies about the formation of splats and their interaction to form a coating were still in their infancy and further investigations would still be needed.

2.5.1 Experimental Work

2.5.1.1 Individual Splat Formation

When a fully melted particle impacted on the substrate, the splat formed might exhibit either a regular disk shape or fingered splashing depending on the particle parameters and substrate conditions. Disc-shaped splats presented better contact of the splat with the underlying surface, which resulted in better adhesion/cohesion and less porosities of the sprayed coatings [2, 6].

In the investigation of the particle flattening and cooling process, Vardelle et al. [103] reported that the extent of the splat spreading was interrupted by its solidification, resulting in various splat shapes. They found the degree of particle flattening to increase and particle flattening time to decrease with an increase in the velocity and temperature of the particle. This accorded with findings of Trapaga et al. [104] and was also confirmed by Leger et al. [105], who provided an explanation for this phenomenon. A rise in particle temperature corresponded to a decrease in the dynamic viscosity of the material, which together with higher particle velocity resulted in higher degrees of flattening. It was well known that the rate of heat transfer from splat to underlying surface was, to a large extent, controlled by the contact area between them and the splat thickness. Leger et al. [105] found that a higher degree of flattening corresponded to a decrease in splat thickness and a larger area of splat surface being in contact with the underlying material. Therefore, cooling rate increased steadily with the flattening degree. For the shape of the splat, it was demonstrated that the degree of splashing increased with particle impacting velocity and tilting angle. Especially for particles impacting with an angle below 60° , the splats became extensively fingered even when the substrate temperature was higher than the transition

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temperature, which would be introduced later, and they could be easily pulled off by a perthometer tip. Correlatively, the coating adhesion/cohesion became very poor.

Recent investigations on the influence of substrate temperature on splat formation revealed the existence of a narrow transition temperature, depending on which the splat shape changed [66, 105]. For substrate above the transition temperature, the splats solidified when flattening was almost completed and thin disc-shaped splats were formed. When substrate temperature was below this temperature, the flattening droplets began to freeze before flattening was completed and part of the liquid splashed away, resulting in extensively fingered splats. Fukumoto et al. [106] found this transition temperature to be characteristic for each combination of particle and substrate material. Vardelle et al. [107] showed that the reason for the particles being formed in disk shapes was that the particle material did not start to solidify during deformation and the liquid remained above the hypercooling temperature. The hypercooling temperature was a function of the melting point of particle materials, the latent heat of melting, and the specific heat of the liquid. Liu et al. showed that a high particle and substrate temperature resulted in a longer solidification time for the splat, producing greater deformation of the droplet. Leger et al. [105] showed the cooling rate for zirconia particles to be higher on a hot substrate than on a cold substrate. Despite the higher cooling rate, the particles impacting on the hot substrate remained as liquid during deformation and formed disc-shaped splats. The shape and adhesion of the splats depended also on the oxides formed on the substrate surface. It was agreed that the growth of oxide layers was promoted by the preheating of substrate. However, there existed different opinions regarding to the role the oxide layer played in the shape of the splat. Bianchi et al. [108] showed that the splats formed on a thick oxide layer (greater than 40nm) were very distorted with fingers of splashed materials at their

periphery no matter if the substrate temperature was above or below the transition temperature. The disc-shaped splats collected on hot substrates were reported to exhibit very low thermal contact resistance. On the contrary, Pasandideh-Fard et al. [109] measured the thickness of the oxide layer formed on the testing substrate and found that it increased with the substrate preheating temperature. They believed that the increasing thickness of oxide layer led to an increase of thermal contact resistance between the splat and the substrate, which was responsible for delaying the onset of splashing and producing the disc-shaped splat. The investigations mentioned above were mostly carried with smooth substrates. On rough substrates, even though the splats on rough substrates were quite different from those collected on smooth surfaces, the same general trends could still be observed.

2.5.1.2 Coating Properties

Ideally, coatings should be formed by the successive building-up of fully melted (although not overheated) particles impacting orthogonal to the substrate with high velocities. However, depending on their respective initial diameter and trajectory, particles in flight experienced quite different accelerations and heating treatments due to the large velocity and temperature gradients of the plasma jet. The portion of unmelted particles could not adhere on the substrate or the deposited layer, and would bounce away which reduced the deposition efficiency of the coating. On the other hand, some partially melted particles were incorporated inside the deposit, which modified the coating microstructure and properties. It was found that the surface roughness of substrate had a great influence on the coating deposition efficiency (DE). A rougher surface enhanced DE due to the improved mechanical adhesion between the substrate and the splat. It was concluded by Kucuk et al. [74] that when spraying YSZ powders, DE was lower for the deposits sprayed (1) with a

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lower torch power, (2) at a larger standoff distance, (3) with an older (worn) torch electrode, and (4) with a higher powder feed rate. In addition, DE also varied with the powder morphology: a hollow sphere YSZ feedstock with high crust density, for instance, yielded a higher DE than crushed YSZ because hollow sphere particles provided a more uniform temperature distribution.

McPherson and Shafer [110] established two types of porosity to be formed in coatings. Coarse porosity was associated with defects in the structure and with incomplete filling of the interstices between the consecutively deposited particles. Fine porosity, with pores ranging from 0.02 to 0.2 μm , were inherent features resulting from entrapped gas, which was often interpreted to represent incomplete contact between the splats and the underlying material. Porosity generally degraded the mechanical properties of materials: for example, brittle fracture was extremely sensitive to defect size and the strength was determined by the largest crack-like defect present. Many other properties such as electrical and thermal conductivity and elastic modulus were reduced by the presence of porosities, which reduced the effective cross-sectional area of the material. The detailed influence of porosity on these properties depended on the pore size and morphology. However, finely distributed porosity tended to improve the thermal shock resistance of brittle materials and especially for the ceramic powder based thermal barrier coatings (TBC), porosity was required to provide thermal insulation and to accommodate operational thermal stress. Cracks were created due to the shrinkage of the splats during the rapid cooling. Bengtsson et al. [111] suggested that if good mechanical contact between the splat and the underlying material was achieved, the initiation and propagation of vertical cracks would act as stress relievers. However, cracks between splats parallel to the substrate surface led to delaminations of the top layer. These were due to failure of the splats to achieve good

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mechanical bonding with the underlying material. Friis et al. [67] systematically investigated the influence of particle in-flight characteristics by controlling the plasma spraying parameters on the microstructure of YSZ coatings produced, especially on the porosities and cracks. They found that particles with higher velocity and surface temperature resulted in dense coatings with less porosity, and substrate temperature and spray angle controlled the vertical cracks or the delaminations. Sampath et al. [112] also found that a higher substrate temperature resulted in a lower degree of porosity, suggesting this to be due to difference in splat shapes and the enhanced contacts between the splats.

The adhesion/cohesion strength actually quantitatively reflected the bonding conditions between the interfaces of substrate-splat and splat-splat. McPherson and Shafer [110] were the first who experimentally confirmed that there was only a limited area in real contact between the interfaces between splats. It was also found that particle impact temperature played a more significant role on the bonding strength of the coatings than the particle velocity. Bianchi et al. reported on a demonstration in which the adhesion/cohesion of the coating was more than doubled when the coating was deposited on a hot substrate rather than on a cold one.

In spite of the various studies carried on investigating the properties of coatings produced under different spraying conditions, the wish to have a clear idea of the relationships between the plasma spraying parameters and the coatings formed has not yet been accomplished. The combination of different powder materials and the plasma spraying system also added to the complexity. As the database was very limited, it was still necessary to collect data on the spray parameters, in-flight particle parameters, and the correlated coating properties.

2.5.2 Numerical Modeling

A fundamental theoretical work on the impact of droplets on the solid surface was reported by Madejski [113]. The model reported by Cirolini et al. [114] was an extension of Madejski [113] formulation with solidification of the splat as Stefan problem. Modification of Madejski [113] model were further made by Zhang [115] to account for the evolution of the solidification front and heat transfer among the molten droplet, deposit layer and substrate. It was concluded that the spreading of large droplets was controlled by viscous dissipation effects and those of the smaller particles were by surface tension.

Some other theoretical works have also been reported on splat formation. Works by Feng et al. [116] Bussmann et al. [117], Nylen et al. [118], and Ghafouri et al. [119] were some among them. In the work reported by Feng et al. [116], a friction limiting condition at the particle-substrate interface was introduced to include the effect of surface roughness. It was found that the radial velocity component of droplet flattening has great influence on the radial velocity distribution. Also the increase in substrate surface roughness resulted in a decrease of radial velocity at the particle substrate interface. This influence resulted in the decrease of flattening degree with an increase of substrate surface roughness.

A model simulating coating thickness, fluid flow and temperature inside the plasma jet, particle temperatures and velocities, and heat transfer between the deposit and substrate was developed by Nylen et al. [118] This was a comprehensive model that connected the process parameters with coating formation through the modeling plasma jet, plasma-particle interaction, and coating formation by the molten particles on the substrate and its experimental evaluation.

Figure 2.1 Schematic illustration of the anodic arc root attachment [3].

Figure 2.2 Classification of the three modes of arc fluctuation according to the voltage waveforms [18].

Figure 2.3 Schematic of the turbulent mixing process of ambient air into the plasma [32].

Figure 2.4 Relative intensity of an object made of tungsten in different temperatures. The gray areas are the two measurement wavelength ranges [54].

(a)

(b)

Figure 2.5 Temperature dependence of the viscosity of (a) Ar-He and (b) Ar-H₂ plasmas at atmospheric pressure [1].

(a)

(b)

Figure 2.6 Temperature dependence of the thermal conductivity of (a) Ar-He and (b) Ar-H₂ plasmas at atmospheric pressure [1].

(a)

(b)

Figure 2.7 Temperature dependence of the electrical conductivity of (a) Ar-He and (b) Ar-H₂ plasmas at atmospheric pressure [1].

Figure 2.8 Image of plasma flame under various chamber pressures [83].

Figure 2.9 Comparison of the new electrodes with the worn ones of (a) cathode, and (b) anode.

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3.1 Plasma Spray System and Powders Used

The plasma spray system employed in the present investigations was a SG-100 (Miller Thermal Inc. USA) plasma torch controlled by the plasma control console from Praxair Surface Technologies (Model 3702). As schematically illustrated in **Figure 3.1**, the overall operation system mainly consisted of the following parts.

- i. Gas supply unit (a): This unit consists of arc gas cylinders and a control unit, which is responsible for the supply of primary gas, secondary gas and carrier gas to the plasma torch according to the required level set by the user.
- ii. Power supply unit (b): Power supply unit is responsible for the supply of arc current and voltage as set by the user. In the present system, only the arc current is adjustable and the voltage value is fixed.
- iii. Powder feeder (c): It consists of powder feeder and powder wheel. The powder feeder operates on volumetric feed principle. Slots in the powder wheel at the base of the canister are filled with powders. Rotation of wheel forces the powder into exit ports and powder feeding rate is controlled by wheel rotation speed. The carrier gas takes the powder from exit port through the powder hose and to the plasma torch.

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- iv. Water booster pump (d): Proper supply of cooling water (distilled water) is a must for the trouble free operation of plasma torch and optimum life of the electrodes are ensured by efficient supply of cooling water.
- v. Plasma torch (e): Plasma torch is the most important component of a plasma spray system, which is responsible for the heating and acceleration of injected powders.

The Praxair SG-100 plasma torch could be equipped with a versatile combination of various cathode, anode, and gas injector models. The torch employed in the present investigations was a subsonic mode consisting a new set of a #1083A-720 thoriated tungsten cathode and a #2083-730 water-cooled copper anode, as shown in **Figure 3.2**. The gas injector ring (#3083-112) had four radial holes to provide a straight injection of plasma gas. The SG-100 plasma torch was designed with internal powder injection, with the opposite two injection ports slightly slanting towards the main-flow direction, as previously shown in **Figure 1.3**. Internal injection of powder provided a higher plasma temperature at the point of powder entry and longer dwell time of the particles within the hot region as compared to external injection. In addition, by injecting the particles within the nozzle, atmospheric effects such as air entrainment would have less influence on the particles during the injection process. The power of the SG-100 plasma torch was provided by a Praxair (Miller Thermal) PS-100 power supply. The power supply was a constant current source for operating the DC torch with a capacity of 1200A/35V. The DC arc was initiated by a Praxair (Miller Thermal) HF-2200 high frequency arc starter. The high frequency starter created a R. F. spark between the cathode and anode, creating an ionized path for the arc column formation. The arc current and gas flow rates (primary gas, secondary gas, and carrier gas) were controlled by the Praxair (Miller Thermal) 3702

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control console and the powder feed rate was controlled separately by a Praxair powder feeder (Model 1264).

In the present experiments, the plasma spray parameters were chosen to favor a “steady” mode of the arc fluctuation in order to minimize the influence of plasma jet instability on the particle in-flight behaviors and the consequent coatings. The plasma torch operated at high current with a straight injection of pure argon as plasma gas, which has been proven to cause little fluctuations of both the voltage and plasma jet. Pure argon also served as carrier gas, inside which the powders were transported from the powder feeder and injected into the plasma torch through either one or two injection ports.

The sprayed powders used in the present experiments were 8wt.% yttria partially stabilized zirconia (YSZ) powders (Norton #204 from Saint-Gobain) and NiCrAlY powders (Ni, 23%Co, 17%Cr, 13%Al, 0.5%Y, Ni-171 from Praxair Surface Technologies) due to their wide applications in producing the thermal barrier coatings (TBC) and many other wear and corrosion resistance coatings. In most of the YSZ material applied coatings, NiCrAlY serves as bond coatings to alleviate the mismatch of the thermal expansion between the top ceramic YSZ coatings and the underlying substrates, and increase the bonding strength between them. Both of these two kinds of powders were fabricated by spray drying and sintering method to assume a near spherical shape but rough surface, as shown by the SEM photos in **Figure 3.3**. Their respective size distribution was shown in **Figure 3.4** and the physical properties were listed in **Table 3-1**.

Table 3-1 Physical properties of the two powders used in the present experiments

	Melting Point ($^{\circ}\text{C}$)	Density (g/cc)	Size Range (μm)	Morphology
YSZ	2750	1.7	< 80	Quasi-spherical
NiCrAlY	1455-1493	3.6	25-80	Quasi-spherical

3.2 Descriptions of the PDA System

Phase Doppler Anemometry (PDA) has received considerable attention over the past 20 years. The success of this method could be attributed to the measurement principles upon which it is based, namely, light scattering interferometry. Light scattering interferometry utilizes the wavelength of light as the measurement scale and, as such, the performance is not as easily degraded as it is for systems using light scattering intensity for the estimation of the particle size. The light wavelength is typically not affected by the spray flow environment, as is the light intensity, for example. In addition, the method does not require frequent calibration. Over the years, the instrument has proven to require only an initial factory calibration. The parameters affecting the measurement which include the laser wavelength and intensity, beam intersection angle, transmitter and receiver focal lengths, and the detector separation seldom change with time.

3.2.1 Laser Doppler Anemometry

For velocity measurements, the Phase Doppler technique is identical with the conventional LDA technique, which was described in detail by Durst et al. [40]. Two parallel laser beams with the same wavelength are focused by the front lens of a laser transmitter to intersect at its focal point, as shown in **Figure 3.5**. It is well known that the intensity of any laser beam has a Gaussian distribution along its cross sectional plane, and the width of the

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beam is usually defined by position where the light intensity is $1/e^2 \cong 13\%$ of the core-intensity. At the focal point the cross section of the laser beam attains the smallest value, and the laser beam is uniquely described by the size and position of this so-called beam waist. Due to the Gaussian intensity distribution, the intersection of the two laser beams, which is also defined as the measurement volume of LDA, is an ellipsoid as indicated in **Figure 3.6**. In this figure, d_f is the beam waist diameter, which is given by,

$$d_f = \frac{4f_l\lambda}{\pi d_e} \quad (3-1)$$

where f_l is the focal length of the front lens in the laser transmit unit, λ is the wavelength of the laser, and d_e is the initial laser beam diameter before exiting from the transmitter front lens. With a laser beam initial diameter $d_e = 2.2$ mm, laser wavelength $\lambda = 514.5 \times 10^{-6}$ mm and $f_l = 310$ mm, the waist diameter $d_f = 0.092$ mm.

The size of the measuring volume can be calculated from the beam waist diameter d_f of the focused laser beams and the intersection angle θ between them:

$$d_x = \frac{d_f}{\cos(\theta/2)}; d_y = d_f; d_z = \frac{d_f}{\sin(\theta/2)} \quad (3-2)$$

where d_x is the height, d_y the width and d_z the length of the measuring volume. Since beam intersection angles are usually small, d_x and d_y are often almost equal, and are sometimes referred to as the diameter of the measuring volume.

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The two laser beams interference at their intersection region to form a fringe pattern. The fringe spacing δ_f can be shown to be proportional to the wavelength λ , and inversely proportional to half of the angle between two incident laser beams $\theta/2$:

$$\delta_f = \frac{\lambda}{2 \sin(\theta/2)} \quad (3-3)$$

From the height d_x of the measuring volume and the fringe spacing δ_f , the total number of fringes can be calculated:

$$N_f = \frac{d_x}{\delta_f} = \frac{d_f / \cos(\theta/2)}{\lambda / 2 \sin(\theta/2)} = \frac{2d_f}{\lambda} \tan(\theta/2) \quad (3-4)$$

The number of fringes applies for a seeding particle moving straight through the center of the measuring volume perpendicular to the fringe direction. If a particle passes through the outskirts of the measuring volume, it will pass fewer fringes and consequently, there will be fewer periods in the recorded signal from which the Doppler frequency can be estimated.

When a particle is passing through the intersection area, it will scatter light and the intensity will change according to the interference fringes. The intensity variation of the scattered light, or the frequency f_D , can be obtained by a photo-detector. The velocity of the passing particle will be proportional to the Doppler frequency f_D and the fringe spacing; i.e.,

$$u = f_D \delta_f = \frac{f_D \lambda}{2 \sin(\theta/2)} \quad (3-5)$$

where u is the absolute velocity of the particle in the direction that is perpendicular to the bisector of the two incident laser beams. The mean velocity U is obtained by,

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$$U = \frac{1}{N} \sum_{i=0}^N u_i \quad (3-6)$$

N is the number of sampled particles. The Root Mean Square (RMS) velocity, U_{RMS} may be expressed as,

$$U_{RMS} = \sqrt{\frac{1}{N-1} \sum_{i=1}^N (u_i - U)^2} \quad (3-7)$$

To obtain good velocity result, a sufficiently high number of fringes in the measuring volume should be ensured. Typical LDA set-ups produce between 10 and 100 fringes. Adding frequency shift by Bragg cell will cause the fringe pattern to “roll” through the measuring volume, increasing or decreasing the number of fringes passed by a seeding particle. If the fringes move towards the movement of the seeding particle, the effective number of fringe-passes will increase, and if the fringes move away from the particle, the number will decrease, corresponding to an increase or decrease in the number of periods in the recorded signal. Through this way, negative velocity can be discriminated from positive velocity, and the range of the measurable velocity will be extended. For a fixed frequency shift f_0 added by Bragg cell, the minimum measurable velocity is down to:

$$u_x > -\frac{\lambda f_0}{2 \sin(\theta/2)} \quad (3-8)$$

where u_x is the velocity component along x -axis. From this equation one can see that, increasing the beam intersection angle will increase the minimum negative velocity.

The maximum measurable velocity is limited by the response-time of the photo-multiplier and the following signal-conditioning electronics. Increasing the beam intersection angle will increase the number of fringes, thus decreasing the fringe spacing. Given the same

velocity, decreased fringe spacing requires the photo-multiplier to respond to the particle transmission in even less time. The characteristics of the photo-multiplier impose a restriction for the minimum response time. Hence an increase of the beam intersection angle will decrease the measurable maximum velocity.

3.2.2 Phase Doppler Anemometry

3.2.2.1 Size Measurement

The principle of the phase Doppler anemometry (PDA) relies on the Doppler difference method used for conventional laser-Doppler anemometry (LDA). For velocity measurements, only one photo-detector is required. By using an extended receiving optical system with two or more photo-detectors, it is possible to measure simultaneously size together with the velocity of spherical particles. For obtaining the particle size the phase shift of the light scattered by refraction or reflection from the two intersecting laser beams is used.

A typical optical set-up of a two-detector PDA system is shown in **Figure 3.7**. The transmitting optics is a conventional dual beam LDA optics. The PDA receiver module is positioned at an off-axis angle α in the $y-z$ plane and consists of a front lens, which focuses exactly at the intersection point of the two laser beams. Portions of laser light scattered by a particle passing through the measurement volume reach the front lens of PDA receiver to become parallel rays, and then pass through a mask that defines the elevation angles of the two photo-detectors (i.e. the angles out of the $y-z$ plane). In this case the mask has two rectangular apertures, which are located symmetrically with respect to the $y-z$ plane at an elevation angle $\pm\psi$. The light rays passing through each aperture are then focused onto a spatial filter, i.e. a vertical slit, which defines the effective length of

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the measurement volume. The effective length of the measurement volume results from the width of the spatial filter l_s (typically about $100 \mu m$) and the magnification of the receiving optics, i.e. the ratio of the focal lengths of the collecting lens to the second lens: $L_s = f_1/f_2 l_s$. Finally, each scattered light is focused onto a photo-detector and guided through the optical fiber to the signal processor.

To explain the operational principle of the PDA, the simple fringe model may be used. Interference fringes in the intersection region of the two incident laser beams are assumed to be parallel light rays [41]. A spherical transparent particle placed into this fringe pattern will act as a kind of lens, whereby the light rays will be projected into space as indicated in **Figure 3.8**. The separation of the projected fringes at a distance f_r from the particle is approximately given by:

$$\Delta s \approx (f_r - f) \frac{\delta}{f} \quad (3-9)$$

where δ is the fringe spacing in the measurement volume as mentioned above and f is an introduce term as an equivalent focal length of the particle,

$$f = \frac{m}{m-1} \frac{d_p}{4} \quad (3-10)$$

with $m = n_p / n_m$ being the relative refractive index of the particle compared with the surrounding medium.

Since small particles are considered and f_r is usually much larger than the particle focal length f , one obtains:

$$\Delta s \approx f_r \frac{\delta}{f} \quad (3-11)$$

the separation of the projected fringes is thus obtained by:

$$\Delta s = \frac{4 f_r \delta}{d_p} \frac{m-1}{m} \quad (3-12)$$

In general it is not possible to measure this spatial separation of the fringes. However, if now two photodetectors are symmetrically placed at f_r with a separation $\Delta s'$ (**Figure 3.8**) the fringes produced by the moving particle will sweep across the two detectors at the Doppler difference frequency. The signals seen by the two detectors will have a relative phase difference given by:

$$\Phi = 2\pi \frac{\Delta s'}{\Delta s} = 2\pi \frac{2 f_r}{\Delta s} \sin \psi \quad (3-13)$$

Using **Equation 3-12** and introducing the fringe spacing, this becomes:

$$\Phi = \pi \frac{d_p}{\delta_f} \frac{m}{m-1} \sin \psi = \frac{2\pi d_p}{\lambda} \frac{m}{m-1} \sin \frac{\theta}{2} \sin \psi \quad (3-14)$$

Here ψ is the elevation angle of one photo-detector measured from the bisector plane of the two incident beams (i.e. the $y-z$ plane in **Figure 3.7** where also the optical axis of the PDA-receiver is located). It should be emphasized that **Equation 3-14** is an approximation valid only for small scattering angle α , which represents the off-axis angle measured from the forward scattering direction. This equation however is very useful in roughly estimating the measurable particle size range for a given system or in performing an approximate design of the optical configuration for small off-axis angles. For the determination of the particle size from the measured phase difference, the required correlations were derived from geometrical optics, which is valid for particle large

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compared to the wavelength of light [120, 121]. The phase of the scattered light is given by:

$$\Phi = \frac{2\pi d_p n_m}{\lambda} \left(\sin \tau - p \frac{n_p}{n_m} \sin \tau' \right) \quad (3-15)$$

where n_m and n_p are the refractive indices of the medium surrounding the particle and the medium of the particle itself. The parameter p indicates the type of scattering, i.e. $p=0, 1, 2, \dots$ for reflection, first order refraction, second order refraction, etc. Moreover, τ and τ' are the angles between the particle surface tangent and the incident and refracted ray, respectively (**Figure 3.9**). For a dual-beam LDA system the phase difference of the light scattered from each of the two beams is given in a similar way:

$$\Delta\Phi = \frac{2\pi d_p n_m}{\lambda} \left\{ (\sin \tau_1 - \sin \tau_2) - p \frac{n_p}{n_m} (\sin \tau'_1 - \sin \tau'_2) \right\} \quad (3-16)$$

where the subscripts 1 and 2 are used to indicate the contributions from both incident beams. For two photo-detectors placed at a certain off-axis angle α and placed symmetrically with respect to the bisector plane at the elevation angle $\pm \psi$, one obtains the phase difference:

$$\Delta\Phi = \frac{2\pi d_p n_m}{\lambda} \beta \quad (3-17)$$

The parameter β , which is also called the geometrical factor, depends on the scattering mode and is given for reflection and refraction by,

Reflection ($p = 0$):

$$\beta = \sqrt{2} \left\{ \left(1 + \sin \frac{\theta}{2} \sin \psi - \cos \frac{\theta}{2} \cos \psi \cos \alpha \right)^{1/2} - \left(1 - \sin \frac{\theta}{2} \sin \psi - \cos \frac{\theta}{2} \cos \psi \cos \alpha \right)^{1/2} \right\} \quad (3-18)$$

Refraction ($p = 1$):

$$\beta = 2 \left\{ \begin{aligned} &\left[1 + m^2 - \sqrt{2}m \left(1 + \sin \frac{\theta}{2} \sin \psi + \cos \frac{\theta}{2} \cos \psi \cos \alpha \right)^{1/2} \right]^{1/2} \\ & - \left[1 + m^2 - \sqrt{2}m \left(1 - \sin \frac{\theta}{2} \sin \psi + \cos \frac{\theta}{2} \cos \psi \cos \alpha \right)^{1/2} \right]^{1/2} \end{aligned} \right\} \quad (3-19)$$

where $m = n_p/n_m$ has been used for convenience and θ represents the angle between the two incident beams. Since the phase difference is a function of p , one expects a linear relation of the correlation between particle size and phase difference only for those scattering angles, where one scattering mode is dominant (i.e. reflection or refraction). Therefore, the values for Φ have been given for these two scattering modes only. Other scattering modes, i.e. $p = 2$, may also be used for phase measurements, especially in the region of back scattering. Such a backscatter arrangement might have advantages with regard to optical access, since both incident beams and scattered light may be transmitted through one window.

By recording now the band-pass filtered Doppler signals from the two photo-detectors, the phase difference $\Delta\Phi$ is determined from the time lag between the two signals as indicated in **Figure 3.10**,

$$\Delta\Phi = 2\pi \frac{\Delta t}{T} \quad (3-20)$$

where T is the time of one cycle in the signal. With **Equation 3-17** it is now possible to determine the particle diameter for a given refractive index n_m and wavelength λ :

$$d_p = \frac{\lambda}{2\pi n_m} \frac{1}{\beta} \Delta\Phi \quad (3-21)$$

From **Equation 3-21** and **Figure 3.10** it is also obvious that only a phase shift between zero and 2π can be distinguished with a two-detector PDA system, which limits the measurable particle size range for a given optical configuration. Therefore, three-detector systems are used, whereby two-phase differences are obtained from detector pairs having different spacing (**Figure 3.11**). This method allows the measurable particle size range to be extended while still maintaining the resolution of the measurement. Moreover, the ratio of the two-phase measurements may be used for additional validation, e.g. the sphericity check for deformable particles such as liquid droplets or bubbles.

3.2.2.2 Particle Concentration and Volume Flux Measurements

PDA has been considered as a useful technique for the concentration and volume flux measurements of spherical particles. Several factors are critical for obtaining reliable results. They are:

1. Accurate measurements of particle velocity, diameter, transit time, etc.
2. Suitable algorithms for flux and concentration calculation as well as auto-calibration techniques of probe volume geometry.
3. Correction techniques for errors caused by an inaccurate validation of the particle signal. Multiple effects (Burst splitting, multi-particle signals, low SNR and so on) cause this variation of the signal number.

A reliable measurement of particle velocity and diameter is the fundamental requirement for concentration and volume flux calculations. Although phase Doppler instruments have been made much more sophisticated to measure single particle size and velocity with satisfactory accuracy, errors of magnitude 100% or greater regarding the volume flux have still been reported. For example, shortly after commercial phase Doppler anemometers became available, Dodge et al. [122] compared the PDA and laser-diffraction techniques in

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the spray produced by a small pressure-swirl atomizer. These researchers reported integrated volume flux measurements that varied from a small percentage of the total flow through the nozzle (measured within 5 mm of the nozzle) to 500% of the total flow through the nozzle (measured 35 mm downstream of the nozzle). Such errors were attributed to several different sources, including improperly sized particles, non-uniform reference area for the measurement or incorrect particle counts due to the poor signal validation.

The most important step in measuring the volume flux by means of PDA method is the determination of the cross-sectional area in the measurement volume. This area, as the reference area for the volume flux, depends not only on the particle size but also on the flow direction of the particles. Gaussian distribution of light intensity in the measurement volume causes a shift of the measured size distribution towards larger particles, compared to the real size distribution, since the probability that larger particles are detected by the system is higher. Signals from smaller particles passing near the center of the measuring volume may have a comparable amplitude as those from larger particles passing through the outskirts of the measurement volume, as shown in **Figure 3.12**. This effect results in an increase of the effective cross-sectional area of the measurement volume with increasing particle size.

The size of the measurement volume is determined by the intersection region of the two incident laser beams and the size of the spatial filter, which images a part of the measurement volume onto the photodetector in the receiving optics. The simplest case is 90° orientation of the receiving optics with respect to the forward scattering direction and the use of a slit as a spatial filter. For one-dimensional flow the particle size-dependent effective cross-sectional area of the measurement volume may be calculated from

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$$A(d_{pi}) = 2r(d_{pi})L_s / \sin \alpha \quad (3-22)$$

where α is the scattering angle of the PDA receiver, L_s is the width of the image of the spatial slit in the receiving optics of the PDA system, d_{pi} is the diameter of the individual particle and $r(d_{pi})$ is the cross-sectional width of the measurement volume depending on the individual particle diameter.

A method for measuring $r(d_{pi})$ for one-dimensional flow was suggested by Saffman [41],

$$r(d_{pi}) = \frac{d_f}{2\sqrt{2}} \sqrt{\ln\left(\frac{S(d_{pi})_m}{S_T}\right) - 2\left(\frac{N_0}{N_f}\right)^2} \quad (3-23)$$

where d_f is the Gaussian waist diameter of the PDA measurement volume, $S(d_{pi})_m$ is the maximum signal amplitude at the center of the burst from a particle passing through the center of the measuring volume, S_T is the trigger level and N_0/N_f is the ratio of the number of zero crossings to validate the burst to the number of fringes in the measuring volume. All these values may be calculated from the parameters of the transmitting optics and the settings of the electronics except $S(d_{pi})_m$, which must be measured.

Substituting **Equation 3-23** into **3-22**, the effective cross-sectional area for each individual particle diameter is given as,

$$A(d_{pi}) = \frac{d_f L_s}{\sqrt{2} \sin \alpha} \sqrt{\ln\left(\frac{S(d_{pi})_m}{S_T}\right) - 2\left(\frac{N_0}{N_f}\right)^2} \quad (3-24)$$

For one-dimensional flow, the local particle concentration is determined by the number of particles passing through the measurement volume per unit time and the effective cross-

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sectional area of the measurement volume for each particle. The particle concentration C_N calculated over a period of time Δt may be expressed by,

$$C_N = \frac{1}{\Delta t} \sum_{i=1}^N \frac{1}{u_i A(d_{pi})} \quad (3-25)$$

where N is the number of particles passing through and being detected, and u_i is the individual velocity of them.

For determining the particle volume flux using the PDA, the number N and the individual diameter d_{pi} of particles passing normal through the reference area $A(d_{pi})$ per unit time are recorded. The following equation gives the expression of particle volume flux normal to the flow direction:

$$F_N = \frac{\pi}{6\Delta t} \sum_{i=1}^N \frac{d_{pi}^3}{A(d_{pi})} \quad (3-26)$$

Equation 3-26 can often be found in a slightly modified form, e.g. if the summation is not performed over each individual particle but rather over groups of particles of similar size:

$$F_N = \frac{\pi}{6\Delta t} \sum_{k=1}^{N_s} \frac{D_k^3 N_k}{A_k(D_k)} \quad (3-27)$$

where N_k is now the number of particles in each size group, i.e. the number/size distribution, and N_s is the number of segments over which the size distribution is computed.

3.2.3 Source of Errors and Uncertainty Levels for LDA and PDA

3.2.3.1 Possible Errors for LDA Measurement of Particle Velocity

It is well known that the principle of LDA velocity measurement is based on the parallel fringe pattern of the intersection region of the laser beams. The prerequisite for accurate measurement of particle velocity is that the two coherent laser beams must intersect at their respective beam waists. Only under this circumstance would the interference produce parallel planes of light and darkness pattern. If the two laser beams do not intersect in the respective beam waists but elsewhere in the beams, the wave fronts will be curved rather than flat, and as a result the fringe spacing will not be constant but depend on the position within the intersection volume. As a consequence the measured Doppler frequency will also depend on the particle position, and it will no longer be directly proportional to particle velocity. If the beams are badly misaligned, the measurement result will be greatly deviated, but if the beams are just slightly misaligned, the errors will be small and have no significant influence on the velocity measurement.

In addition, successful detection of a passing particle by the LDA requires that enough number of fringes should be crossed by the particle in order to validate a measurement. Thus, a particle passing through the measurement volume at a large angle may not be recorded. However, this problem could be obviated by introducing a fixed frequency to one of the two laser beams with the aid of the Bragg cell to make the fringes move relative to the flow.

Another source of errors inherited with the LDA measurement is the validation bias. Validation bias is a broadening of the definition of the filter bias. Filter bias is any deviation of the data processing system from a flat frequency response over the possible

range of velocity measurements. This bias is a result of band-pass filtering the Doppler signal too close to valid signal frequencies, making those velocities less likely to be measured. Validation bias includes filter bias, but is expanded to include processor biases. It is defined as any deviation from flat validation response for the entire processing system, over the range of possible velocity measurements.

Table 3-2 lists the accuracy of the LDA velocity measurement for some of the uncertainties arising from the sources of errors mentioned above.

Table 3-2 Accuracy of the velocity measurement by LDA

Statistical	Accuracy of Signal Processor	Velocity bias
$\pm 0.5\%$	$\pm 0.05\%$	$\pm 1\%$

(Taken from: www.dantecmt.com)

3.2.3.2 Possible Sources of Error in PDA and Their Minimization

Since its first introduction, the Phase Doppler technique has been analyzed by numerous researchers [44, 49, 50, 123] in order to determine its possible error sources. Since the principle of the particle velocity measurement is identical with that of the LDA, which has been extensively examined previously [40], emphasis was primarily put on investigating the accuracy of the particle diameter, concentration and volume flux measurements. Early evaluations of the measurement capabilities involved the use of monodisperse streams of droplets of a known size, directed through the measurement volume of the PDA. The agreement of the measurements using the Phase Doppler instrument with the expected size was well within one percent. Tests were also conducted in which the monodisperse drop streams were measured with sprays located in the intervening paths of the transmitted

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beams and the light scattered to the receiver. Such tests were also carried out with turbulent flames in the optical paths and the tests showed only insignificant effects of the optical disturbances produced by the sprays and flames [44]. Measurement comparisons have been made with other means for characterizing the sprays and with estimates of the droplet size based on the aerodynamic relaxation, droplet terminal velocity, as well as the use of other strategies [124]. Measurements have also been made of sprays interacting with highly turbulent flows [125]. These studies, as well as being informative for the dynamics of such turbulent two-phase flows, provided confidence in the measurement method as well since the drop response to the gas phase flow could be used as a means for estimating the accuracy of the data when measuring in complex spray flow environments. The phase Doppler method has been applied in hostile environments such as dense sprays produced by gas turbine and rocket injectors and in highly turbulent combustion environments. The instrument has generally performed well in these difficult applications.

Although the phase Doppler anemometry (PDA) is a well established technique for measuring size and velocity of spherical particles, some difficulties are encountered with the measurement of large particles, in particular when the system is set up to detect refractively scattered light, as is usually the case in typical spray applications. Large particle means that the particle diameter is larger than 20% of the diameter of the measurement volume [123]. In such cases erroneous size measurements may lead to a significant reduction of accuracy in estimating volume weighted flux and concentration, since the volume is proportional to the third power of diameter. Two effects have been identified as potentially leading to incorrect size measurements in such applications: Trajectory effect, or Gaussian beam defect [44, 49, 123] and Slit effect [50, 126]. Both effects arise when the PDA system is set up to receive refractively scattered light, but

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instead receives reflectively scattered light. Those signals are then also processed with the phase-diameter relationship based on refraction and thus lead to incorrect size measurements.

Trajectory dependent scattering effect occurs in phase Doppler measurements as a result of the non-uniform or Gaussian intensity distribution of the incident laser beams in the measurement volume. The problem arises when, for certain particle trajectories, the magnitude of the reflected rays from the particle become comparable to that of the refracted rays. For example, in the forward scattering angles, reflection effects become significant when large particles pass through the edges of the measurement volume far from the optical receiver, seen from **Figure 3.13**. It is well known, however, that in phase Doppler anemometry the linearity of the phase versus size calibration curve depends upon the existence of a single dominant scattering mode (for example, either reflection or refraction). Therefore, when the reflected rays and the refracted rays achieve comparable magnitudes, the mutual interference of the two causes the phase versus size calibration curve to deviate from linearity. This leads to particle sizing errors. The problem of trajectory dependent scattering becomes more significant when the phase Doppler technique is used to probe dense spray environments, for example rocket injector sprays, where the size of the probe volume needs to be decreased in order to avoid the presence of multiple particles within the measurement probe volume. This, in turn, causes the particle-to-probe diameter ratio to increase and the system becomes more susceptible to the errors associated with trajectory dependent scattering.

In a phase Doppler anemometer (PDA), a slit is used in the receiving optical unit in order to have a well-defined length of the measuring volume for flux measurements and

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concentration measurements. A small effective length of the measuring volume also enables the instrument to measure under high particle concentration conditions. A slit serves as a spatial filter. It is commonly believed that the length of the measuring volume is the width of the image of the slit projected onto the intersecting laser beams and only when a particle is inside this region will it be detected.

However, the slit is not a perfect spatial filter, and it can cause erroneous size measurements when particles pass through the measuring volume along certain trajectories and the length of the measuring volume, over which particles are detected, can be much longer than expected. This unexpected effect of the slit on the PDA measurements is termed the slit effect.

In **Figure 3.14**, the refracted light and the reflected light have different paths. Light will thus be detected even when the particle center is outside the slit aperture. When the particle is at a positive z location as shown in **Figure 3.14**, the refracted light is blocked by the slit but the reflected light can still be detected. Since the phase of the reflected light is different from that of the refracted light, the size measurement is incorrect when the refraction mode is selected for the size measurements. This effect is very similar to the Gaussian beam defect, but the error can be more significant and can also occur with small particles.

The effect of non-uniform light intensity in the measurement volume of a phase Doppler system has been studied by a considerable number of researchers over the past years. Meanwhile, different techniques have been suggested to minimize or eliminate the Gaussian beam defect [44, 50] and possible approaches to avoid the slit effect have also

been proposed [50, 126]. Among these proposals there are three approaches that seem to be effective and technically feasible:

1. Use modified optical arrangements.
2. Reject bursts influenced by the Gaussian beam effect or slit effect according to the inconsistency of signal phases in different detector pairs.
3. Reject bursts influenced by the Gaussian beam effect or slit effect according to signal amplitude, burst length or signal-to-noise ratio (SNR). The level for the rejection can be set as a function of measured size.

In general, both the Gaussian beam defect and the slit effect are caused by the non-uniform light scattering when particle passes through the probe volume along certain trajectories. Gaussian beam defect caused by the mixing of the reflected light and the refracted light is of comparable magnitude, while the slit effect occurs when the refracted light from a particle is blocked by the slit in the receiving optics but the reflected light or a high order refracted light can still be detected. Thus it could be seen that techniques applied to minimize or eliminate the Gaussian beam defect can also be used to overcome the problems of slit effect. In the above-mentioned three approaches to improve the size measurements, the first method is used widely. After realizing the inability of conventional PDA system in solving these problems, different optical configurations have been proposed. All of them can be considered as extension of the conventional PDA, with the aim to suppress or eliminate the Gaussian beam defect and the slit effect so as to achieve more accurate sizing measurements. Later in this report, all of the modified configurations to the conventional PDA are termed as Extended PDA systems, as schematically illustrated in **Figure 3.15** [127]. For the detailed introduction of extended PDA systems, please refer to **Appendix I**.

Table 3-3 The accuracy of velocity and diameter measurements by the PDA.

	Range	Accuracy	Resolution
Velocity	< 500 m/s	± 0.5%	± 0.03%
Diameter	> 0.5 μm	± 0.5%-± 1%	1.41°

(Taken from www.dantecmt.com)

3.2.4 The PDA Configurations for the Present Experiments

The Phase Doppler Anemometry (PDA) used in the present experiments was a two-dimensional system from Dantec Inc.. Laser beam generated by a 2W (Model Spectra-Physics Stabilite 2017) Argon-ion laser, whose main excitation wavelengths were 488nm (blue light) and 514.5nm (green light), passed through a 60X24 FiberFlow transmitter probe to be split into a pair of blue laser beams and a pair of green laser beams by the interior prism. The plane formed by the pair of green laser beams was perpendicular to the ground to measure the particle impacting velocity u along the main flow direction and the plane formed by the pair of blue laser beams was horizontal to the ground to measure the particle transversal velocity. The PDA transmitter probe was mounted with a 50X57 front lens of 310mm focal length, which focused the two pairs of laser beams at the same point. The intersection point of the laser beams, i.e. the measurement volume of the PDA, was observed by a 57X80 DualPDA receiver probe mounted with a 55X12 Beam Expander of expander ratio 1.98, with the aid of a 60X117 Conical Adapter. The front lens of the PDA receiver probe was a 50X58 standard one with focal length 500mm. The front lens of the PDA receiver probe worked as a collimator, which collected the scattered light from the beam intersection when a particle passed through, and created a beam of parallel light. This beam then passed through an aperture plate. The DualPDA receiver probe was originally designed for the Dual Mode PDA setup, which consisted of a conventional PDA and a planar PDA configurations. However, it could be easily converted to a three-detector FiberPDA by utilizing the FiberPDA aperture plates in the DualPDA receiver probe, as

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adopted in the present experiments due to the more robust measurement of FiberPDA. The parallel light generated by the front lens of the PDA receiver probe passed through a FiberPDA aperture plate (Mask A, as shown in **Figure 3.16**), which divided the parallel light beam into three segments. Corresponding to the three segment apertures of the FiberPDA aperture plate were four focusing lenses segments cemented together into a composite lens, which was the original part of DualPDA receiver probe and the focal length of these segment focusing lenses was 125mm, as also shown in **Figure 3.16**. Each of these segment lenses focused a portion of the parallel light beam to form an image of the intersection volume on one of four slit-shaped spatial filters in front of an optical fiber. The width of the spatial filters was $100\ \mu\text{m}$. The part of the image that fell on the slit itself corresponded to the measurement volume. Only the light from the measurement volume could be allowed to pass through the optical fiber to reach the photo-multipliers. These light components were then guided through four multi-mode optical fibers, each representing a specific viewing angle. The four fibers ran in a single cable leading to the 58N81 DualPDA Detector Unit. Before entering the detector unit, the light transported inside the four fibers passed through four corresponding interference filters. The function of these narrow-band interference filters was to let pass only the desired wavelength and block away unwanted wavelength components. As shown in **Figure 3.16**, the light components corresponding to the segment focusing lenses U_1 , U_2 , and U_3 passed through three corresponding green interference filters, while only the light component of V_1 passed through one blue interference filter. The four light components passing through their respective interference filters were then lead to four photo-multipliers inside the detector unit to be converted to electrical signals, i.e. the Doppler Burst. The computer interface socket connected the 58N80 MultiPDA signal processor to the 58G130 PDA interface board in the computer. After calibration, the signals of U_1 , U_2 , and U_3 were used to

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calculate the particle impacting velocity along the main flow direction u and particle size d , and the signal of V_1 was used to calculate the transversal velocity component perpendicular to the main flow direction v . In plasma spray process, the particle transversal velocity v was much smaller than the particle impacting velocity u and had little effect on the microstructure of coatings formed. Thus, they were of little importance and were neglected in the present investigations. The phase differences of each of the two signals pairs, i.e., U_1/U_2 and U_1/U_3 , were compared with each other for the particle diameter and sphericity check according to the preset criteria. Only particles conforming to the allowable tolerance of diameter and sphericity variations were measured while the rest of the particles that failed to satisfy these criteria were rejected.

Velocity measurement based on the frequencies of the Doppler Bursts is a well-established technique, and the optical alignment of PDA is mainly aimed at securing a linear dependence of particle diameter on the phase difference of the signals. To ensure the two laser beams intersect at their beams waists to generate the parallel fringe pattern, each of the beams will be first adjusted carefully so that they could intersect just at the focal point of the transmitter front lens. This was achieved with the aid of a focus-alignment target from TSI.

As demonstrated in the expression of the relationship between the phase difference and particle diameter mentioned above, the sensitivity and range of the PDA changed with any of the beam intersection angle θ , scattering angle α , and elevation angle ψ . In practice, the three angles can not be chosen freely. The aperture plate mounted inside the PDA receiver probe (Mask A of FiberPDA here) actually defined the elevation angles of the viewpoints and the measurable particle size range. The intersection angle influenced the measurable

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particle velocity range. Keeping other settings constant, the intersection angle of the two laser beams could not be too small to ensure there are sufficient fringes in the measurement volume. However, too large intersection angle would cause particles with too high velocity to be rejected due to the restriction of the response time of the photo-multipliers and the signal processor. Generally speaking, the scattering angle α was the most critical parameters governing the measurement accuracy when applying the PDA in detecting transparent materials like water droplets or glass beads. A linear relationship between the phase difference of the Doppler Bursts and particle diameters only existed when the PDA receiver probe was placed in a scattering angle where only one light scattering mode dominated. However, the particle materials employed in the present experiments were all non-transparent, and only reflected light from these particles could be used for the PDA measurement. Thus, the scattering angle was selected to obtain an acceptable signal-to-noise ratio and from practical considerations of the measurement situation. The PDA parameters chosen for the present experiments were remained constant as tabulated in Table 3-4.

Table 3-4 PDA Configuration Settings

<i>Optical Configurations</i>	<i>Parameters</i>
Laser Power	1.0 [Watts]
Wavelength of Laser Beam	514.5, 488.0 [nm]
Beams Intersection Space	20 [mm]
Beams Intersection Angle θ	3.69°
Polarization of Beam	Perpendicular
Off-Axis Angle α	110°
Focus Length of Transmitting Unit Lens	310 [mm]
Focus Length of Receiving Unit Lens	500 [mm]

Figure 3.1 Schematic diagram of typical plasma spray system.

Figure 3.2 The cathode (bottom) and anode (top) of the plasma torch used in the present experiments.

Figure 3.3 SEM photos of the original (a) NiCrAlY and (b) YSZ powders.

Figure 3.4 Size distributions of the (a) NiCrAlY and (b) YSZ powders.

Figure 3.5 Measurement principle of backscattered LDA system (taken from www.dantec.com).

Figure 3.6 Measurement volume formed by intersection of two laser beams.

Figure 3.7 Typical optical configurations for a two-detector Phase Doppler Anemometer (PDA).

Figure 3.8 Schematic illustration of the relationship between particle diameter and the separation spacing of the two photo-detectors.

Figure 3.9 Schematic illustration of a ray refracted by a particle.

Figure 3.10 Determination of the phase shift from the two band-pass filtered Doppler signals [121].

Figure 3.11 Phase-size relations for a three-detector phase Doppler system [121].

Figure 3.12 Schematic illustration of the increase of the effective cross-sectional area of the PDA measurement volume with increasing particle size.

Figure 3.13 Illustration of Trajectory effect / Gaussian beam effect.

Figure 3.14 Illustration of Slit effect.

Figure 3.15 Schematic illustrations of Extended PDA systems: (a) Three detector FiberPDA, (b) Modified Standard PDA, (c) Planar PDA, (d) DualMode PDA.

Figure 3.16 The basic layout of the 57X80 DualPDA probe coupled with FiberPDA Mask.

Chapter 4

Measurements of Plasma-Sprayed Particles Using PDA

4.1 Introduction

For characterization of the plasma spraying process, measurements of particle temperature, velocity, and size in the environment of high speed, chemically reacting, and ionized gas streams always pose a great challenge. Non-intrusive optical diagnostics must be used primarily due to the hostile environment. Combining the Laser Doppler Anemometry (LDA) and Phase Doppler particle sizing technique, the PDA has proven its capability and reliability in obtaining simultaneously the velocity and size of individual spherical droplet or solid particle in polydisperse flow environments. Originally developed as a method for measuring gas turbine atomization, air-fuel interaction, and liquid fuel combustion, the PDA has evolved to become a standard diagnostic technique for spraying characterization of the size and velocity of sprayed drops and other spherical particles in a wide range of applications. However, it is only in recent years that the PDA technique was utilized in characterizing the plasma sprayed solid particles [51], and the literatures addressing the usage of PDA in the plasma spraying conditions are relatively rare [52, 53].

Nowadays, most of the PDA systems utilized in various researches and laboratories are mainly commercial products, which undergo factory calibration and require no future calibration for the users. Proper configuration of the PDA settings is the primary consideration to apply successfully this technique in characterizing the particle behavior in various research fields. It is well known that the performance of the PDA technique

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depends greatly on the physical properties of particles, flow conditions and the PDA configurations. Appropriate selection of the PDA parameters with respect to the specific application ensures reliable measurements, which is especially important for the application of the PDA in detecting transparent materials. In such case, the Gaussian beam effect and slit effect are the main sources of errors [49, 50]. Both effects cause the PDA receiver to detect the mixture of different scattered light components or wrong light component. However, the PDA seems to have obvious advantage in measuring the plasma sprayed particles since the powder materials employed for plasma spraying are commonly non-transparent. Under this condition, the mixing of different scattering modes of light from particles would be naturally avoided. Thus, the receiver probe of the PDA can be positioned within the complete scattering angle range, apart from near forward scattering position where diffracted light is present.

Without the problem of having reflected and refracted light components together, the primary considerations for applying the PDA technique in the plasma spraying process are thus the local particle density and the particle morphology. The PDA technique is theoretically a single particle counting method for spherical particles with smooth surface, which means that any occurring of more than one particle in the measurement volume as well as the deviation of particle morphology from the optimum conditions (spherical and smooth-surface) will inevitably affect the measurement accuracy. In the plasma spraying process, the local particle density and the possibility of the multi-particle coincidence occurred in the PDA measurement volume can be numerically evaluated with the aid of Poisson statistics equations. As for the morphology of the sprayed particles, even those fabricated by atomization and spray drying to especially obtain a spherical shape may not achieve the perfect spherical shape and smooth surface. Signals of light scattered by the

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particles with irregular shape or rough surfaces may be misinterpreted by the signal processor, leading to erroneous particle size calculation. This phenomenon is especially important for the volume flux measurement by the PDA, which relies predominately on the percentage of large particles measured since it is proportional to the third power of the particle diameter. In this case, the misinterpretation of a small particle as large one would cause significant error in the final calculated value of the volume flux. To the best of the author's knowledge, these problems arising from the application of the PDA in measuring plasma sprayed particles have never been considered before.

In the following sections, the possibility of multi-particle coincidence phenomenon occurring in the plasma spraying process for the PDA measurement will first be analyzed. The sensitivity of the light signals detected by the PDA to the shape and surface roughness of the plasma sprayed particles will be evaluated experimentally through observing the waveforms of the Doppler bursts from different kinds of particles. This will be followed by the investigations on the reliability and accuracy of the PDA measured particle diameter and volume flux.

4.2 The Possibility of Multi-Particle Coincidence

The theory of the phase Doppler technique states that the measurement of a single sphere illuminated by two interfering laser beams can be made quite accurately [46-49]. Single-particle generators can be used to demonstrate this and, in fact, have been used to calibrate the equipment.

However, the practical spraying consists of a poly-dispersion of particle sizes. This causes uncertainties for two reasons: one is related to the data acquisition process, and the other is

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related to analysis required to account for the poly-dispersion. The former is due largely to the limits associated with the technique. First, since the PDA instrument is a single-particle counter, it is required that only a single-particle be present in the measurement volume during the measurement. In a mono-dispersed particle stream, not only is a single-particle size present, but also the particles are generated with known spacing, which eliminates (assuming the particle spacing exceeds the waist of the focused overlapping beams) the possibility of multiple particles in the measurement volume. In a poly-dispersed spraying, the random behavior and size/velocity correlations due to varying momentum and body forces requires that some probability of multiple particles in the measurement volume exists.

Since the PDA technique is based on a single particle counting method, it requires that only one particle passes the measurement volume at one time. This sets a limitation on the particle number densities that can be accommodated with the method. Bachalo et al. [128] concluded that the particle number density affected on PDA performance as following:

- Single particle counters can only successfully measure one particle at a time.
- More than one particle in the sampling region at one time should cause the rejection of the event.
- The probability of more than one particle being present in the sampling volume at one time may be predicted using Poisson statistics.
- As the particle number density increases, the measured number density will increase to a maximum, and then decrease due to increasing coincident occurrences.

Figure 4.1 shows the case of particles arriving at the measurement volume at a rate of more than one particle is residing in the measurement volume at one time. To reduce the

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probability of such coincident occurrences when measuring such dense spraying, the measurement volume size must be optimized.

The trace of the signals for a dilute spraying where little or no coincident occurrences are produced is shown in **Figure 4.2 (a)**. In the case of a dense spraying where coincident occurrences are more probable is shown in **Figure 4.2 (b)**. It can be seen that the two signals overlap in the case of two-particle coincidence inside the PDA measurement volume. The signal processor may either identify this latter case as a multiple particle occurrence and reject both signals, or only process the first signal and ignore the second. It is also possible that a weighted average of the phase shifts between the two signals is recorded.

The particles are assumed to be distributed randomly and homogeneously in space and have a number density η . The PDA works well in low number density sprayings ($\eta < 10^3 \text{ cm}^{-3}$), while it is fraught with problems in dense, poly-disperse sprayings where the total number density can typically reach 10^5 cm^{-3} [128], which is often encountered in the high-pressure liquid rocket injectors. In this case, the measurement volume should be configured to minimize the multi-particle occurrence.

The measurement volume can be estimated by [129],

$$V = \frac{\pi d_f^2 L_s}{4 \sin \alpha} \quad (4-1)$$

where d_f is the $1/e^2$ beam waist diameter, L_s is the apparent slit width and α is the scattering angle. The apparent slit width is equal to the slit width multiplied by the magnification factor of the receiving optics. For a scattering angle of 110° , a beam waist of

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92 μm , a slit width of 100 μm and a magnification factor of the receiving optics 1.98, the measurement volume is about $1.4 \times 10^{-6} \text{ cm}^3$. Treating the particles as infinitesimally small, the probability of finding n particles within the measurement volume can be determined by [129],

$$P(n) = \frac{(V\eta)^n}{n!} e^{-(V\eta)} \quad (4-2)$$

where V is the measurement volume and η is the particle number density. Thus,

$$\frac{P(n+1)}{P(n)} = \frac{V\eta}{n+1} \quad (4-3)$$

For the above listed configuration with $\eta = 10^5 \text{ particles/cm}^3$, the ratio, $P(2)/P(1) = 0.07$, which means that the probability of finding two particles in the measurement volume is only 7 percent that of finding one. For the plasma spraying process, the particle number density is commonly less than $10^5 / \text{cm}^3$, so the multi-particle coincident effect should be relatively small.

In reality, the PDA instrument may not be able to reject all the coincident occurrences. Logic systems including redundant phase measurements, signal to noise ratio checks, and signal amplitude measurements are all incorporated into the better PDA instruments to prevent erroneous measurements. However, it is still possible that the coincidental events may be recorded. For example, if a large particle and a small particle are residing in the measurement volume at one time, the signal from the large particle will dominate and may be successfully measured. In other cases, particles of nearly equal size may pass with approximately the same velocity (Doppler frequency) and phase, and they may also be recorded as a single passing particle.

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4.3 Assessment of the PDA Diameter Measurement for the Plasma-Sprayed Particles

Based on the Mie scattering theory, the measurements of a single smooth sphere by the Phase Doppler technique have been proven to be quite accurate [41, 47, 49]. The application of PDA technique in the plasma spraying field has contributed a lot to the understanding of the spraying behavior and future amelioration of the plasma torch design. However, according to the author's knowledge, among the meager existing literatures addressing the utilization of the PDA in characterizing the plasma spraying process, no one had ever attempted to assess the potential errors for the PDA size measurement. They may be even more pronounced when PDA is applied to the complex plasma spraying dealing with the solid particles whose initial shape and surface conditions are commonly not spherical and smooth. In the following section, efforts were devoted to evaluating the reliability and accuracy of the PDA in measuring different size group of the NiCrAlY powder samples subjected to the plasma jet and discussing how the surface properties of the particles would affect the measurements.

4.3.1 Signals of Light Generated from Non-Spherical Particles

Before proceeding to the experimental investigation, a close look into the fundamentals of the PDA size measurement enables a better understanding of the phenomena encountered when using the PDA to measure the plasma sprayed particles. As geometrically illustrated in **Figure 4.3**, for a given illumination wave and a detector position, each scattering mode, e.g. reflection or first-order refraction, traversed along the unique path to each detector after impinging and interacting with the particle. For particles larger than the wavelength of light, as in the case of powder particles used for plasma spraying, both the amplitude and the phase of the incident waves vary across the diameter of the particle. Effectively the

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particle projects only certain parts of the incident waves onto the photo-detector. Thus only certain areas of the particle surface are involved in defining signal properties. The position and size of the receiving aperture define the position and size of these interaction areas. The area of the first interaction of the incident wave with the particle surface is known as the “incident point” and the source area where the scattered light reflected from the particle surface is called “glare point”. The size of the incident areas/points and glare areas/ points is proportional to the size of the receiving aperture [121].

If two illumination waves are used, then at least two glare points will exist for each scattering mode. These glare points can be viewed as coherent light sources directed towards the detector, which will produce interference fringes in the far field, similar to the classical Yong’s fringe experiment. These fringes will move in space according to the particle movement through the intersection of the illuminating waves and according to which scattering mode is being considered. The fringe movement or the frequency of modulating measured on a detector yields the particle velocity, as with the laser Doppler technique. Thus, two detectors separated in space will each detect a modulated signal as the interference fringes cross their apertures; however, a phase shift results due to the spatial separation of the detectors. The phase shift can be related to the spatial fringe spacing and to the distance between glare points on the particle surface through the geometric parameters of the optical system.

For reflection mode of the PDA as adopted in the present experiments, the incident point and the glare points are coincident. Thus, the position of the reflection points on the particle surface is determined by the detector position and beams orientation. Particle diameter is interpreted from the trace of the reflection points on the particle surface, based

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on the local radii of curvature, as shown in **Figure 4.4**. Since the particle diameter validation criterion only checks the consistency of the curvature for part of the particle surface, one may conceive of non-spherical particles being validated. One example would be the case of a non-spherical particle passing through the measurement volume in such a way that a spherical or nearly spherical region of the particle surface scatters the light to be detected by the PDA receiver while regions with other curvatures do not scatter light in the directions of the detectors. The perceived size of such a particle would then correspond to the curvature of the spherical region facing the detectors. In this case, if the particle is deformed into a prolate spheroid, it will be sized as a smaller spherical particle. On the contrary, particles in the form of oblate spheroids will be sized as larger spherical ones, as schematically shown in **Figure 4.5**. Therefore, the PDA diameter measurement is significantly influenced by the orientation of the particle relative to the position of the PDA receiver.

The principles of the signal generation are basically valid for both spherical and non-spherical particles. The PDA technique measures the particle diameter through the local curvature information obtained from entire glare points on the particle surface when reflection mode of the PDA is considered. However, a complete determination of the shape and structure of a more irregular particle with rough surface becomes increasingly difficult because of the limited number of sample glare points on the surface of particle. It was found experimentally that the response of a PDA system deviated from the theoretical prediction when surface roughness is present. The deviation is more pronounced with increasing surface distortion. When the radii of curvature at any point of distortion on the particle surface are comparable to the incident wavelength, the second-order reflection at the particle surface must be considered [130], as shown in **Figure 4.6**. In this case a

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double-scattering ray emerges from a point. The far-field fringe pattern formed by the scattered lights from the irregular particle surface thus becomes distorted, which introduced unpredictable fluctuations to the phase difference of the Doppler signals detected between different photo-detectors and lead to random errors to the measured particle diameter.

To demonstrate the influence of the particle surface condition on the light scattering and the signals detected by the PDA receiver, waveforms of the Doppler burst were recorded by an oscilloscope for water droplet, plasma sprayed NiCrAlY particle and plasma sprayed YSZ particle respectively. The water was sprayed through an atomizer with very low pressure. The PDA measurement volume was put under the orifice of the atomizer at a standoff distance of 10 mm, where the sprayed water droplets were believed to assume perfect spherical shape due to the surface tension effect. NiCrAlY and YSZ powders were plasma sprayed under their respective common operation conditions in which a coating with acceptable deposition efficiency could be produced according to our previous experience [131, 132]. The PDA measurement volume was located at the standoff distance of 80 mm from the exit of the plasma torch and on the mean trajectory of the particle plume for the two kinds of powders respectively, where the possibility that particles could be melted was higher. Particles in their molten state would naturally exhibit spherical shape and smooth surface, which were proven by the SEM photos of the two kinds of powders with and without subjecting to the plasma jet heating shown in **Figure 4.7**. The powders were sprayed directly towards a basin of water, after cooling down, were collected and dried for subsequent SEM observation. It was obvious that the particle sphericity and surface condition improved after the particles had been plasma sprayed. The

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improvement was much more pronounced for the NiCrAlY powders since its melting point was only about half that of the YSZ powders and thus much easier to be melted.

With the oscilloscope connected on the signal output ports of the PDA signal processor, the waveforms of Doppler bursts were recorded for the three kinds of particles mentioned above respectively. For each kind of particles, at least 20 waveforms were recorded, but only the most representative one that was not only successfully detected by the PDA but also validated for the particle size measurement was selected for demonstration, as shown in **Figure 4.8**. It was obvious that the Doppler bursts induced by the water droplet and the plasma sprayed NiCrAlY particle were quite regular, while the one induced by the YSZ particle had a more irregular distribution. The phenomenon may be attributed to the reason that the former achieved perfect spherical shape and smooth surface when passing through the PDA measurement volume, while the YSZ particle recorded was not fully melted due to its relatively higher melting point.

The waveform of Doppler burst recorded by the oscilloscope is actually a reflection of the current/voltage pulse from the photodetector, which contains the frequency and phase information relating to the particle velocity and size to be measured. However, the photocurrent is always accompanied by noise. Noise is essentially any amplitude deviation of the signal from the analytic form. Sources of noise are manifold, including stochastic noise coming from the photodetector and electronics (shot noise, white noise, Johnson noise, dark current, etc.) as well as from the scattering process itself. The primary source of noise is the photodetector shot noise, which is a fundamental property of the detection process. The interaction between the optical field and the photo-sensitive material is a quantum process, which unavoidably impresses a certain amount of fluctuation on the

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mean photocurrent. In addition there is mean photocurrent and shot noise from undesired light reaching the photodetector. For a particle with irregular shape or rough surface, the reflected light detected by the photo-detector of the PDA may be comprised of light rays coming from inconsistent glare areas on the particle surface which represent different curvatures. Thus, the current contribution to the photo-detector is added with random phases, and the resulting Doppler signal envelope and phase will fluctuate. In this case, the PDA signal processor will normally estimate phase difference of the signals as weighted average value, which makes the deviation of particle diameter measurement inevitable.

Generally speaking, the signal-to-noise ratio (SNR) should be as high as possible to ensure an over-weight of the desired signal over the undesired noise. However, in the current application of the PDA in measuring plasma-sprayed particles, the particles under measurement are in extremely high temperature and will spontaneously emit light by themselves. Both the light emitted by the particles due to the high temperature and the random photons emitted by the plasma surround them are sources of noise among many others. In this circumstance, the amplitude of the noise might be in the same order of magnitude as that of useful signal, i.e. SNR around 0 dB. Thus, a SNR much higher than 0 dB may cause the over-rejection of the particle measurement, especially for smaller particles from which the laser light reflected will be much lower, and reduce the data validation rate. In the current experiments, the range of SNR settings recommended in the PDA software manual is -6 to 3 dB and the actual SNR was chosen within this range to achieve the highest data validation rate.

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For the three-detector PDA configuration adopted in the present experiments, the phase difference corresponding to each detector pair gives the information about the curvature over a certain arc of the particle surface. Due to the different elevation angles, the phase difference/diameter relations for the three detector pairs are different, the closely spaced pair yielding a relation which is less steep, as shown in **Figure 4.9**. Two of the three phase differences are linearly independent and can be used for two independent particle diameter estimations. The third one can be used as a particle sphericity validation because the sum of all the three phase differences should be zero. For a perfect spherical particle with smooth surface, two such pairs of photo-detectors should correspond to the same particle diameter d_p . However, in practice some tolerance must be accepted in the agreement of the two separately measured diameters due to the existence of the noise in the signals and the deviation of particle morphology from perfect sphericity and smooth surface. One validation criterion used in the PDA software is the maximum allowable deviation from perfect sphericity. Thus, if the difference between the two separately measured particle diameters Δd exceeds a certain limit set by the user, the particle diameter will be rejected. In the present experiment, the maximum allowable $\Delta d / d_p$ was set as $\pm 20\%$.

A two-dimensional matrix is generated by the PDA software associated with the two phase differences Φ_{12} and Φ_{13} , as shown by a typical plot exported from the PDA software in **Figure 4.10**. The sphericity validation criteria are satisfied only if the diameter value falls within the shaded areas. If these criteria are not satisfied, the particle will be rejected as demonstrated by the dots outside of the shaded areas. A non-acceptance band for phase difference $\Phi_{13} > 260^\circ$ are implemented as out of range (hatched area in **Figure 4.9**) due to visibility consideration.

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4.3.2 Diameter Measurements for NiCrAlY Powder Samples in Different Size Ranges

In order to evaluate the reliability and accuracy of the PDA size measurements, especially the influence of particle shape and surface roughness, NiCrAlY powders were sieved to different size groups, as listed **Table 4-1**. The samples for each size group were tested under four different one-port spraying conditions: cold spray, plasma spraying under 500A, 700A and 900A of arc current respectively. The detailed spraying parameters were listed in **Table 4-2**. The three current values were chosen to represent three levels of plasma jet energy, which rendered the in-flight particles different degree of heat treatment. Since the PDA does not distinguish between opaque powder materials that are either light absorbing or totally reflecting, NiCrAlY powders were selected due to their relatively lower melting point. The particles in their molten state would naturally assume a spherical shape and smooth surface due to the surface tension effect, which is especially desirable for the PDA measurement. For the original gross NiCrAlY powders, particles sprayed under the 700A arc current were expected to experience the optimum heating and acceleration inside the plasma jet with most powders being melted (but less occurrence of particle overheating and evaporation). Under the condition of lower power input (500A arc current) to the plasma torch, the particles would be heated to a lesser extent and the percentage of the unmelted particles would increase. Increasing the plasma arc current to 900A would further increase the number of particles being melted, however the number of overheated and evaporated particles would also increase at the same time. Other than the arc current settings, a primary gas flow rate of 72.23slm, a carrier gas of 5.22slm and powder feeder rate of 20 g/min were maintained constant for all the plasma spraying conditions. The PDA configurations were listed previously in **Table 3-4**. The PDA transmitter and receiver probes were mounted on a computer-controlled 3-D traversing rig. For cold spraying, the measurement volume of the PDA was placed just at the exit of the powder injection port.

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For plasma spraying, the experimental rig was illustrated in **Figure 4.11 (a)**. The plasma torch was oriented upside down to facilitate the PDA measurement and powders were injected inside the plasma torch through one powder injection port. The measurement volume of the PDA was traversed along the particle injection direction (radial direction) at a standoff distance of 80mm from the exit of the torch nozzle. The interval of measurement point was 1mm. At each measurement location, at least 2000 samples were collected in order to satisfy the statistical requirement. At the point where PDA measurement acquired the highest data rate, i.e. the mean trajectory of the particle plume, the PDA measured particle size distribution was obtained.

Table 4-1 The individual size range of the four groups of NiCrAlY samples

	Group A	Group B	Group C	Group D
Size Range (μm)	35-43	43-57	57-65	65-75

Table 4-2 The operation conditions for spraying the NiCrAlY sample groups

Cases	Arc Current	Plasma Gas		Carrier Gas		Powder Feed Rate	
	<i>Ampere</i>	<i>psi</i>	<i>slm</i>	<i>psi</i>	<i>slm</i>	<i>rpm</i>	<i>g/min</i>
1	N/A	N/A	N/A				
2	500			40	5.22	2.0	30
3	700	80	72.23				
4	900						

Figure 4.12-15 show the SEM picture of the original sieved NiCrAlY powders and their size distributions characterized by the PDA under the four spraying conditions mentioned above for each of the four size groups respectively. NiCrAlY powders being cold sprayed remained their original properties with irregular shape and rough surface. In addition, many particles were in the form of agglomerates, which consisted of many bumps that could be regarded as mini particles attached to their surfaces. In this circumstance, the

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PDA diameter validation rate, which is defined as the ratio of the particles satisfying the PDA sphericity check for size measurement to all the particles that could generate signals detected by the PDA, were extremely low for the PDA diameter measurement of all the four size groups, typically lower than 10%. However, even for those validated particle diameters by the PDA software as shown in the histograms of the size distribution for the cold spray, they were obviously deviated significantly from the original size ranges. The reason has been elaborated in **Section 4.3.1**. Depending on the orientation of the particle relative to the position of the PDA receiver probe and the glaring area on the particle surface, the scattered light might be interpreted by the photo-detectors to only represent the curvature of some local areas on the particle surface. For example, if the little bump on the particle surface happened to scatter light that was detected and satisfied the PDA sphericity criterion, the PDA measured diameter would be that of the little bump instead of the particle itself. On the contrary, if the surface area of an elongated particle scattered light to the PDA receiver, the PDA would give a diameter value larger than the actual diameter of the particle. Many other random scatterings from the particle surface might also be accidentally accepted for the particle diameter measurements. Thus, for the PDA measurements of the cold-sprayed particles, a large portion of them would give misleading diameter readings even if they happened to pass the particle sphericity check.

The NiCrAlY particles being melted in the plasma jet obtained spherical shape and smooth surface due to the surface tension effect, as early demonstrated in **Figure 4.7**. In comparison with the histograms of the PDA measured particle diameter distribution in the cold spraying condition, it was obvious that the PDA measured plasma sprayed particles fell well within their respective size range, especially for the smaller size groups. Under this circumstance, the PDA diameter validation rate rose to over 75% in most of the plasma

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spraying conditions except for **Group D**. It should be reminded that the mechanical sieving of powders might not be able to exactly separate the particles to their respective size groups. There would always be smaller particles appearing in the larger size groups. In addition, an irregular particle falling within a size group during the sieving process might exhibit a different diameter after being melted to achieve a spherical shape. Thus, one could expect that some of the diameters measured by the PDA might be slightly out of the expected range.

For smaller particles in size **Group A**, the PDA measured diameters exhibited very narrow distribution. The PDA diameter validation rate in this case was as high as 85% even for the lower power input condition (500A arc current). This phenomenon was attributed to the reason that smaller particles were heated and melted rapidly inside the plasma jet due to their smaller mass and lower specific heat capacity. Therefore, the thermal energy of the plasma jet under lower current condition was still high enough to melt the smaller NiCrAlY particles. Most of the smaller particles passing through the PDA measurement volume were in their molten state, which was most desirable for the PDA to accurately calculate their diameters. The broadening of particle diameter distribution for the PDA measurements of size **Group B** compared with those of **Group A** was evident, which demonstrated a tendency of an increased number of particle diameters being miscalculated by the PDA software. The miscalculations of particle diameters in **Group B** were predominantly caused by the unmelted particles, as proven by the fact that increasing the arc current level from 500A to 900A led to obvious narrowing of the diameter distribution and a good agreement of the PDA measured diameters with the original particle size range, at the same time accompanied by an increase of the PDA diameter validation rate. Increasing the power level increased plasma enthalpy correspondingly, which in turn

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resulted in an overall improvement of the heat treatment of the in-flight particles and increased number of particles being melted when passing through the PDA measurement volume. The PDA measured particle diameter distributions were broader and broader for the size **Groups C and D**, indicating more and more particles being miscalculated by the PDA software. Although it was still clearly exhibited that the diameter distribution became narrower with increased power input for the histograms of **Group C**, this phenomenon was almost not distinguishable for the largest size range of **Group D**. In this circumstance, even the higher power input could not bring significant increase to the number of melted particles. This was because that larger particles had higher initial momentum upon injection into the plasma jet, which made them easier to penetrate the core region of the plasma jet rapidly without undergoing sufficient heating. In addition, the high specific heat capacity of the larger particles required longer heating time for them to be melted.

4.4 Correlation between the PDA Measured Particle Volume Flux and the Volume of Actual Deposits

By observing the diameter distribution histograms for the plasma sprayed NiCrAlY powders presented in the previous section, it could be concluded that the PDA could measure the diameter of melted or surface-melted particles accurately. However, random errors arose for measuring the unmelted particles and it was found that many of the PDA measured diameters were out of the size range of the sample groups. Accompanied by an obvious drop of the diameter validation rate in the PDA software, this phenomenon was especially pronounced for the PDA measurement of larger particles under lower power input condition, in which most of the particles could not be melted.

As mentioned earlier, the original gross NiCrAlY powders passed through the 80 μm sieve completely. Taking into consideration the 20% diameter tolerance set in the PDA software,

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the maximum particle diameter should not exceed $100\ \mu\text{m}$ theoretically. It is also unlikely to have particle annexations taking place during the particle in-flight, which could be readily proven by the SEM pictures of the plasma sprayed powders. Thus, it could be concluded that all the particle diameters measured by the PDA to be larger than $100\ \mu\text{m}$ were most probably miscalculations of the smaller unmelted particles. These overestimated particle diameters might not play a significant role when evaluating the average particle diameter at a measurement point since they usually occupied only a small fraction of all the diameters being measured. However, they would lead to erroneous results when they were included in the particle volume flux measurement by the PDA software.

In this section, the PDA measured particle volume flux was compared with the volume per unit time of the actual deposit. YSZ powders with particle diameter lower than $80\ \mu\text{m}$ were utilized in this investigation and the spraying conditions were listed in **Table 4-3**. The measurement volume of the PDA was traversed throughout the entire cross-sectional plane of the particle plume at the standoff distance of 80mm from the nozzle exit and covered a $20\ \text{mm} \times 20\ \text{mm}$ area with $1\ \text{mm}$ interval between any two neighboring measurement points. At each location, the PDA measurement stopped upon satisfying either of the two criteria: (1) 2000 validated particles for both velocity and diameter measurements and (2) a cut-off measurement time of 20 seconds. Integration of the particle volume flux at each measurement location over the whole cross-sectional plane gave the total volume per unit time of the passing particles that were injected inside the plasma torch and at the same time validated by the PDA. Corresponding to the plasma spraying parameters for each case of the PDA measurements, powders were then sprayed directly onto a substrate placed also at the same standoff distance of 80mm stationarily for a given period of time to form a deposit. As shown in **Figure 4.16**, the plasma torch was mounted on the accurately

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computer-controlled 3-D traversing rig mentioned previously for supporting the PDA transmitter and receiver probes. Firstly, the plasma spraying was initiated outside of the substrate until the plasma jet reached a steady state. The plasma torch was then traversed rapidly to the front of the substrate, stopped there and kept spraying onto the substrate for 5 seconds to form a deposit before it was finally moved away. One of the deposits produced was shown in **Figure 4.17**. Each deposit was later brought under CMM machine for surface profile scanning to determine the thicknesses across the whole surface of the deposit. By dividing these thicknesses with the period of spraying time (5 seconds here) and then integrating them over the whole bottom area of the deposit, the volume of the actual deposit per unit time could thus be obtained, which was then comparable with the integration value of the PDA measured volume flux over the entire cross-sectional plane of the particle plume.

Table 4-3 The plasma spraying conditions of the YSZ powders for the comparison of the PDA measured volume flux with the volume of the actual deposit

Cases	Number of Injection Port	Arc Current <i>Ampere</i>	Plasma Gas		Carrier Gas		Powder Feed Rate	
			<i>psi</i>	<i>slm</i>	<i>psi</i>	<i>slm</i>	<i>rpm</i>	<i>g/min</i>
a	One Port	850	60	56.85	40	5.22	2.2	20
b							3.3	30
c	Two Ports	850	60	56.85	30	4.19	2.2	20
d							3.3	30

4.4.1 Algorithms of the PDA Volume Flux Measurement

Particle volume flux was defined as the total volume of the particles passing through a unit area per unit time, as has been elucidated in **Chapter 3**. For the particle volume flux calculation at certain measurement location, the PDA software recorded the diameter and velocity information of each validated particle passing through the measurement volume,

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and at the same time determined the effective cross sectional area of the PDA measurement volume corresponding to that particle size. The particle volume flux was then derived as a cumulative function of the individual particle velocity, diameter and effective cross sectional area for all the validated particles within a certain period of time.

Here the “validated particles” were accentuated since only the particles satisfying all the validation criteria of the PDA measurement could be included in the particle volume flux measurement. Many particles might be able to induce signal detection by the PDA receiver, while they would still be rejected if they could not pass the signal validation procedure. Whereas signal detection referred to determining whether a particle was in the measurement volume or not, validation was used to decide whether the estimated signal parameters, e.g. frequency, phase difference, transit time, arrival time, etc., were trustworthy. The PDA validation procedures were commonly consisted of the signal-to-noise ratio (SNR) validation, phase difference validation, sphericity validation, amplitude validation, transit time validation and arrival time validation. SNR validation is performed by comparing the estimated signal SNR in dB with some pre-defined acceptance threshold. Phase difference validation and sphericity validation procedures have been previously introduced, as shown in **Figure 4.9** and **Figure 4. 10**. Amplitude validation is to eliminate the Gaussian beam effect and the slit effect to avoid unwanted scattering orders becoming dominant in the received signals. The transit time validation could be used to eliminate the data points that arise due to large particles being evaluated as small ones, a typical situation when the slit effect is present or when multiple particles are recognized as one particle [121].

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During the PDA measurement process, the PDA parameters should be selected carefully to ensure the highest data validation rate, which was defined as the number of particles validated for both the velocity and diameter measurement over the total number of particles inducing the signal detection by the PDA. In our experiments for plasma spraying of either NiCrAlY powders or the YSZ powders, velocity measurements based on the PDA technique could easily achieve validation rate higher than 95 percent under the optimum PDA configurations. However, size validation rate observed lower than 40 percent at certain locations, especially at the peripheral region of the plasma jet, was not rare. As demonstrated in the previous section, the rejection of particle diameter was mainly caused by non-sphericity and surface roughness of the particles in the plasma spraying condition. Therefore, the particles that passed the velocity validation but failed the diameter validation would still be rejected for the volume flux calculation.

Several experiments have been made to evaluate the accuracy of the volume flux measurement of the PDA by comparing them with the results derived from other instruments or methods under the same experimental conditions. Almost all the previous works done for this comparison used water droplets or glass beads. Comparisons were made between the PDA volume flux measurements with that obtained by the patternator. The volume flux in the patternator was calculated by measuring the collected volume of water, the elapsed time and the sampling area. The volume flux obtained by the PDA should be further calibrated and compensated relevant to the rejection of particle data to reduce the measurement error. For the quasi one-directional particle flows, the volume flux obtained by the PDA was compensated simply by multiplying the inverse of the size validation rate, assuming that the rejected particles had the same size percentage distribution as the accepted ones and they were rejected mainly because they could not

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satisfy the sphericity criteria. However, the patternator could not be applied in plasma spraying since the extreme high temperature in the plasma jet precluded any use of intrusive devices. At the same time, if the compensation of the particle volume flux would be beneficial for other applications, the case was totally different for the PDA volume flux measurement in the plasma spraying process. As it was well known that the coating/deposit was formed by the successive building-up of fully or partially melted particles impacting on the substrate or previous deposited layers. Solidification of each particle was believed to be completed before the next particle hit at the same location due to the extremely rapid cooling of particles at impact. Thus, the portion of unmelted particles could not adhere on the substrate or the deposited layer and would bounce away. On the other hand, the unmelted particles would most probably be rejected by the PDA software for failing to satisfy the diameter validation. Therefore, it could be concluded that it would most probably be those particles validated for the volume flux calculation by the PDA that contributed to the actual formed deposit/coating.

According to the above discussions, the value of the PDA measured particle volume flux at each measurement point corresponded to the actual thickness of the sprayed deposit at the same location. The equation for the particle volume flux F_N ($mm^3/mm^2/s$) measurement along the plasma sprayed particle flow direction is given by,

$$F_N = \frac{\pi}{6\Delta t} \sum_{i=1}^N \frac{d_{pi}^3 \cos \gamma_i}{A(d_{pi})} \quad (4-4)$$

where d_{pi} and γ_i were the individual particles diameter (m) and trajectory angle (*Degree*).

Here $\gamma_i = \tan^{-1}(v_i / u_i)$, u_i and v_i were the individual particle velocity components along the main flow direction and the transverse direction respectively. $A(d_{pi})$ was the size-dependent effective cross-section of measurement volume along the main flow direction

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(z -axis here) in mm^2 and it was pre-determined by a set of algorithms for different size classes by the PDA signal processor. N was the number of particles detected at each measurement location. This equation may be written as,

$$F_N = \frac{\pi}{6} (C_N u \sqrt{1 + (\tan \gamma)^2}) D_{30}^3 \quad (4-5)$$

where u and γ were the mean particle velocity (m/s) and mean trajectory angle (*Degree*) at each measurement location and C_N was the particle concentration ($1/mm^3$), which was defined as,

$$C_N = \frac{1}{\Delta t} \sum_{i=1}^N \frac{\cos \gamma_i}{u_i \sqrt{1 + (\tan \gamma_i)^2} A(d_{pi})} \quad (4-6)$$

and D_{30} was the mean volume diameter (m) given by,

$$D_{30} = \left(\frac{1}{N} \sum_{i=1}^N d_{pi}^3 \right)^{1/3} \quad (4-7)$$

In the plasma spray process, the ratio $\gamma = v/u$ was typically lower than five percent. The nearer the measurement point was to the centerline of the plasma torch, the smaller the particle movement trajectory biases from the main flow direction. In this case, $\tan \gamma$ was approximately zero and $\cos \gamma \cong 1$. Thus, **Equation 4-6** could be simplified to,

$$C_N = \frac{1}{\Delta t} \sum_{i=1}^N \frac{1}{u_i A(d_{pi})} \quad (4-8)$$

And,

$$F_N = \frac{\pi}{6} u D_{30}^3 C_N \quad (4-9)$$

From **Equation 4-9**, it was obvious that the particle volume flux was predominately influenced by the particle diameter with the third power relationship between them. Miscalculation of the particle diameter would have the most direct and significant effect on the particle volume flux. As mentioned earlier in the beginning of this section, the

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misinterpretation of smaller unmelted particles as larger ones would lead to great deviation to the volume flux evaluation since it was the larger particles that contributed most to the particle volume flux calculation. For example, for a smaller particle of $30\ \mu\text{m}$ in diameter and a larger one of $60\ \mu\text{m}$ in diameter included in the volume flux calculation, the contribution made by the larger one would be 8 times of the smaller one neglecting the minor effect of different velocity and different effective cross sectional area of the PDA measurement volume for different particle size. Thus, if the diameter of that smaller particle ($30\ \mu\text{m}$) was miscalculated as $150\ \mu\text{m}$ for certain reason, the error for volume flux would be around 125 times of the value that it should be.

4.4.2 Comparison Results

To evaluate the correlation of the PDA measured volume flux with the volume per unit time of the actual deposit, comparisons had been made between these two cases under the same plasma spraying conditions. The detailed experimental procedure has been introduced above. The PDA configurations were the same as those listed in **Table 3-4**. In order to investigate the variation of both the PDA measured volume flux and the volume of the actual deposit with different plasma spraying conditions, YSZ powders were injected into the plasma torch through one transversal internal injection port (**Figure 4.11 a**) and two opposite internal injection ports (**Figure 4.11 b**) respectively under different plasma spraying parameters as listed in **Table 4-3**. The difference between the influence of these two powder injection schemes and the plasma spray parameters on the in-flight particle characteristics and the properties of the plasma sprayed coatings will be discussed further in **Chapter 5** and **Chapter 6**.

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As mentioned earlier, the maximum diameter of the YSZ powders used in the present experiments was lower than $80\text{ }\mu\text{m}$. Taking into account the 20% diameter tolerance preset in the PDA software, the particle diameter supposed to be accurately measured by the PDA should not exceed $100\text{ }\mu\text{m}$. Thus, all the particle diameters measured larger than $100\text{ }\mu\text{m}$ could be assumed to be miscalculations of the smaller particles by the PDA software. In order to assess the influence of these miscalculated particle diameters on the PDA measured volume flux, a modified volume flux value was introduced here by filtering all the data of particle diameters measured by the PDA that were larger than $100\text{ }\mu\text{m}$ for each measurement location. After interpolating the individual volume flux values at all the measured points, the particle volume flux distributions across a $20\text{mm}\times 20\text{mm}$ area were plotted for the original and modified particle volume flux respectively, which were subsequently compared with the thickness distribution of the actual deposit per unit time. Based on the Simpson's 3/8 Rule, the total volume per unit time of the YSZ powders was calculated by integrating the individual particle volume flux values at each measurement location for the PDA measurements and the individual thickness values for the actual deposit over their respective bottom area.

Figures 4.18-21 show the surface and contour maps of the PDA measured original and modified volume flux distributions as well as the thickness distribution of the actual deposit. It could be observed that for all the four spraying conditions, the PDA measured original volume flux distribution had irregular surface while the modified volume flux distribution exhibited a smooth surface profile that was closer to that of the actual deposit. This phenomenon indicated that the existence of the miscalculated particles larger than $100\text{ }\mu\text{m}$ included in the PDA measured original volume flux value at each measurement location greatly affected the overall volume flux distribution. **Table 4-4** listed the overall

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integrated volume of the original particle volume flux distribution, modified particle volume flux distribution and the deposit thickness distribution for the four spraying conditions as listed in **Table 4-3**. It could be noticed that the volume of the actual deposit was lower than the total volume obtained from the original particle volume flux distribution but higher than that obtained from the modified particle volume flux distribution. This phenomenon was reasonable due to the fact that the smaller particles being miscalculated as larger ones would lead to significant increase to the volume flux value at each measurement location and consequently the total volume obtained from the original volume flux distribution. On the other hand, it was impossible for the PDA to accurately measure every melted particle that would contribute to the deposit formation. The omission of these melted particles would inevitably reduce the total volume of particles measured by the PDA. Furthermore, the PDA measured volume flux distribution did not take into consideration the porosities that would be inherited in the actual deposit. According to the previous literatures, the total porosity ranged from a few percent to about 20 percent, depending on the particle velocity and temperature. Thus, the total volume obtained from the modified volume flux distribution would be expected to be smaller than that of the actual deposit.

Table 4-4 Integrated volume of the deposit, PDA measured original particle volume flux, and PDA measured modified particle volume flux.

Spraying Conditions	Deposit Volume (mm ³)	Integrated Volume of the Original Particle Volume Flux Distribution		Integrated Volume of the Modified Particle Volume Flux Distribution	
		Value (mm ³)	Ratio to Deposit Volume	Value (mm ³)	Ratio to Deposit Volume
a	13.199	18.507	140.22%	11.204	84.88%
b	18.624	33.596	180.39%	14.758	79.24%
c	15.372	23.628	153.71%	11.194	72.82%
d	23.483	40.126	170.87%	19.337	82.34%

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4.5 Summary

Based on the Poisson statistics equations, the possibility of the multi-particle coincidence occurred in the PDA measurement volume was numerically evaluated, which demonstrated that the phenomenon of two or more particles appearing simultaneously in the PDA measurement volume was rare under the plasma spray condition, and it only played a neglectable effect on the PDA rejection of particle.

The influence of particle morphology on the signals of the scattered laser light detected by the PDA receiver has been qualitatively evaluated from the waveforms of the Doppler burst recorded for the water droplet, plasma sprayed NiCrAlY particle and YSZ particle respectively. It was observed that the waveform of the Doppler burst was very sensitive to the surface condition of the particle. The more spherical and surface-smooth the particle was, the more regular the waveform of the Doppler burst would be, which ensured accurate determination of particle diameter. This finding was supported by comparisons of the PDA diameter measurements of the NiCrAlY powder samples in known narrow size ranges under different spraying conditions. For smaller particles plasma-sprayed with higher power input, most of the particles passing through the PDA measurement volume were in their molten state, which corresponded to a higher diameter validation rate for the PDA measurement. The particle diameters were accurately recorded by the PDA measurement, which could be readily proven by the fact that the PDA validated particle diameters fell well within the original size range of the samples. Plasma spraying of larger particles under lower power input conditions would lead to insufficient heating of the particles and an increased number of particles that would not be melted. In this circumstance, the PDA diameter validation rate dropped considerably. Although the PDA measured mean particle diameter was still in the range of the original samples, the diameter distribution broadened

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significantly, which indicated an increased number of particles whose diameter had been miscalculated.

The reliability of the PDA measurement was further evaluated by comparing the PDA measured YSZ particle volume flux with the volume of the corresponding actual deposit. It was demonstrated that even though the volume flux distribution originally obtained from the PDA measurement was greatly affected by the existence of the miscalculated particles, especially those smaller unmelted particles that were mistakenly recorded as larger ones, the modified volume flux distribution obtained by filtering those larger miscalculated particles exhibited a close surface profile and total volume value with those of the corresponding actual deposit. The variation of the deposit local thickness distribution and the total volume with the plasma spraying parameters could also be approximately reflected by the PDA measured modified particle volume flux distribution.

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Figure 4.1 Schematic showing the problem of coincident particle occurrences in the sample volume.

Figure 4.2 Doppler burst signals for (a) noncoincident and (b) coincident occurrences.

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Figure 4.3 Illustration of the incident points and glare points of the light on the particle surface [121].

Figure 4.4 The PDA measured curvature along the particle surface for diameter measurement [121].

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Figure 4.5 Schematic showing the PDA diameter measurement for the deformed drops.

Figure 4.6 Geometry of the second-order reflection point on the particle surface.

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Figure 4.7 The SEM images of the powders before and after being plasma sprayed for (a) NiCrAlY powders and (b) YSZ powders.

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Figure 4.8 The waveforms of the Doppler burst detected by the PDA for (a) water droplet, (b) plasma sprayed NiCrAlY particle and (c) plasma sprayed YSZ particle.

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Figure 4.9 Particle diameter measurements by the two pair of photo-detectors of the PDA.

Figure 4.10 Comparison between the two phase differences for particle sphericity check.

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Figure 4.11 Experimental setup for the PDA on-line monitoring of the in-flight behavior of particles injected through (a) one transverse injector and (b) two opposite injectors.

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Figure 4.12 The SEM image of the original NiCrAlY before spraying in size **Group A** (**Table 4-1**) and the PDA measured particle diameter distribution for (a) cold spraying, plasma spraying under (b) 500A, (c) 700A and (d) 900A arc current.

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Figure 4.13 The SEM image of the original NiCrAlY before spraying in size **Group B** (**Table 4-1**) and the PDA measured particle diameter distribution for (a) cold spraying, plasma spraying under (b) 500A, (c) 700A and (d) 900A arc current.

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Figure 4.14 The SEM image of the original NiCrAlY before spraying in size **Group C** (**Table 4-1**) and the PDA measured particle diameter distribution for (a) cold spraying, plasma spraying under (b) 500A, (c) 700A and (d) 900A arc current.

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Figure 4.15 The SEM image of the original NiCrAlY before spraying in size **Group D** (**Table 4-1**) and the PDA measured particle diameter distribution for (a) cold spraying, plasma spraying under (b) 500A, (c) 700A and (d) 900A arc current.

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Figure 4.16 The arrangement of plasma torch and substrate to produce the deposit stripes and coatings.

Figure 4.17 The YSZ deposit produced by stationary spraying onto a substrate.

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Figure 4.18 The surface and contour maps of the (a) original PDA measured particle volume flux, (b) modified PDA measured particle volume flux, and (c) actual deposit thickness distributions at 80 mm for YSZ powders sprayed under condition a in Table 4-3.

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Figure 4.19 The surface and contour maps of the (a) original PDA measured particle volume flux, (b) modified PDA measured particle volume flux, and (c) actual deposit thickness distributions at 80 mm for YSZ powders sprayed under condition b in Table 4-3.

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Figure 4.20 The surface and contour maps of the (a) original PDA measured particle volume flux, (b) modified PDA measured particle volume flux, and (c) actual deposit thickness distributions at 80 mm for YSZ powders sprayed under condition **c** in **Table 4-3**.

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Figure 4.21 The surface and contour maps of the (a) original PDA measured particle volume flux, (b) modified PDA measured particle volume flux, and (c) actual deposit thickness distributions at 80 mm for YSZ powders sprayed under condition d in Table 4-3.

Chapter 5

Influence of Plasma Spraying Parameters on Particle In-Flight Characteristics

5.1 Introduction

Investigations into the plasma spraying process have emphasized the importance of understanding the in-flight characteristics of particles before impacting onto the substrate to form a coating. Particles temperature and velocity at impact and the manner they are piling up determine the coating microstructure and properties. It is well known that when the injected gas-powder flow mixture interact with the plasma jet, heat exchanges between the cold mixture and the hot plasma jet cause changes in the particle velocity, diameter and also temperature distribution. The manner and duration of this interaction depends on the injected particle momentum, particle loading rate and plasma jet properties. The injected particle momentum is the product of particle mass and injection velocity, and the latter is primarily provided by the carrier gas flow. For a given plasma jet (arc current, plasma gas flow rate, etc.), an in-flight particle would have different trajectories depending on the particle injection conditions, which may include the carrier gas flow rate, the position and tilting angle of the powder injection port (or injector).

When the carrier gas flow rate was too low, the particle would not penetrate into the plasma jet but would be swept away from the plasma flow due to its insufficient initial momentum. However, when the carrier gas flow rate was too high, the particle would pass through the plasma jet without undergoing sufficient heating. The optimum trajectory

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would be for the particle to penetrate inside the plasma jet and travel along the hottest zone of the plasma jet. In this case, the particle would reach a higher surface temperature and experience better acceleration, which was most desirable for the particle to be deposited on the substrate with higher adhesion strength. Thus, to achieve the higher deposition efficiency and better coating properties, the average momentum of the injected powder should always be adjusted according to the momentum of the plasma jet. This would ensure that majority of particles experienced a longer residence time in the hottest zone of the plasma jet.

In order to determine the influence of the various spraying parameters on the microstructure and properties of the plasma sprayed coating, the most direct way is to change the parameters of interest one by one and examine the resulting coatings. However, the metallurgical preparation of the coatings for microstructure and other properties analysis can be very tedious and time-consuming. More importantly, there is usually no linear or straightforward relationship between most of the individual spraying parameters and the coating properties. In addition, many uncontrollable conditions such as the wearing state of the electrodes contributed much to the uncertainty of the correspondence between the parameters set in the control board of the plasma spray system and the consequent coating properties. Therefore, with the development of the optical diagnostic devices, more and more efforts have been devoted to monitoring and controlling the particle in-flight parameters such as the particle velocity, temperature and diameter. Efforts to establish the relationships between these in-flight particle parameters and the resulting coating properties are of great importance. Knowing the impacting particle parameters under the optimal operation conditions for producing the desired coating, any deviation could be observed and compensated. Particle velocity measurements based on the Laser Doppler

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technique have been widely recognized as a reliable method. Unfortunately, particle velocity information could not be used to predict the resulting coating properties since there exists no direct and linear relationship between them [67, 73-75]. Particle temperature at impact is believed to be the most important parameter determining the coating properties. However, measurements of particle temperature, especially for the plasma sprayed particles, have always been a challenge. Optical temperature measurements are commonly based on the well-known Planck's law of radiation. According to the Planck's law, an object emits electromagnetic radiation, and the intensity and wavelength distribution of the radiation depends on the temperature of the object (the detailed introduction of the particle temperature measurement can be found in **Appendix II**). Most optical diagnostic devices for particle temperature determination are nowadays based on two-color pyrometry. The two-color pyrometry determined the particle surface temperature through measurements of the ratio of the light radiation fluxes emitted by an object in two separate wavelengths, and assumes the particle is a gray body, i.e. the emissivity does not depend on the wavelength. However, this gray-body assumption of the particles is not always tenable, especially for the metallic materials. If the ratio of the emissivities of the material in the measurement wavelength ranges is known, the error could be corrected. Unfortunately, the wavelength and temperature behavior of most plasma spraying materials are usually unknown. Moreover, it has also been demonstrated that the plasma light scattered by the particles and the evaporation cloud around the particle surface may lead to miscalculation of the actual particle surface temperature [54, 56].

In this chapter, PDA technique has been utilized to systematically investigate the influence of the four user adjustable parameters of the SG-100 plasma spraying system, namely the current level, plasma gas flow rate, carrier gas flow rate and the powder feed rate, on the

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in-flight YSZ particle characteristics. The former two parameters are believed to have the most pronounced effect on the plasma jet properties, while the latter two determined the powder injection momentum. The in-flight particle velocity, diameter, concentration and volume flux distributions characterized by the PDA are compared not only among different settings of spraying parameters, but also among different arrangements of powder injector(s). Powders will be injected inside the plasma jet internally and externally through either one injector or opposite two injectors respectively. The powder injection schemes investigated in the present experiment are schematically shown in **Figure 5.1** and the dimensions are illustrated in **Figure 5.2**. For the internal injection of powders, the powder injector(s) was/were located at 12mm upstream position from the torch nozzle exit and perpendicular to the axis of the plasma torch. For the external injection of powders, the powder injector(s) was/were located at 9mm downstream position from the nozzle exit, either perpendicular to the axis of the plasma torch or in 75° counterflow direction. For the powders injected through opposite two injectors, the powder-gas mixture flowing out of the powder feeder was divided almost evenly into two streams by a Y shape splitter, and injected into the plasma torch through the two injectors respectively. The measurement volume of the PDA was traversed along the radial direction, i.e., the x -axis as previously shown in **Figure 4.11**, with an interval of 1 mm at the standoff distance of 80 mm from the nozzle exit for all the spraying conditions. At each location, the PDA measurement stopped upon satisfying either of the two criteria: (1) 2000 validated particles for both velocity and diameter measurements and (2) a cut-off measurement time of 20 seconds. Measurements were stopped at the two edges of the particle plume where the validated data rate of passing particles detected by the PDA approached zero. The distance between the two edge locations was treated as the width of the particle plume at the 80 mm standoff distance.

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As discussed in the previous chapter, the PDA measurement was subjected to some random errors, which would significantly affect the PDA measured particle volume flux values. However, the modified PDA measurement through filtering those particles whose diameter was calculated by the PDA software to be larger than the expected maximum diameter value would provide a more accurate characterization of the in-flight melted or partially melted particles. Thus, in the following investigations of the particle in-flight behaviors, all the raw data exported from the PDA software will all be post-processed to filter the particles whose diameter recorded is larger than the expected maximum diameter.

5.2 Numerical Modeling Results of the Plasma Flow Field

Before starting the PDA measurements, numerical modeling of the plasma jet temperature and velocity distributions were performed for spraying conditions listed in **Table 5-1**. The objective of this simulation work is to provide a benchmark for understanding the importance of particle injection location and tilting angle regarding to the plasma jet. Depending on the particle initial injection location, where the plasma jet velocity and temperature are different, their in-flight trajectories inside the plasma jet would be varied significantly. A three-dimensional model was used via the commercial software Fluent V6.02. Plasma flow was represented as a chemically inert gas with temperature dependent thermodynamics and transport properties in Local Thermodynamics Equilibrium (LTE). Operation of the torch was assumed to be in steady state with negligible gravity effect. The mathematical model was based on the fully elliptic Navier-Stokes system of differential equations with the Reynolds method of averaging the time dependent equations, together with the standard $\kappa - \varepsilon$ model of turbulence. The detailed description of the modeling procedures can be found in [92].

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Table 5-1 Operating and boundary conditions for plasma spraying simulation.

Operating Condition	Value
Current Input	900 A
Voltage	35 V
Torch Efficiency	67 %
Heat Source	$4.4178 \times 10^{10} \text{ W/m}^3$
Arc Gas Flow Rate (Argon Gas)	$1.9474 \times 10^{-3} \text{ kg/s}$
Turbulence Intensity at Arc Gas Inlet	4.83 %

Figure 5.3 shows the simulated temperature and velocity distribution of the plasma jet on the *xz* plane, with the schematic illustration of the position of powder injectors and the mean trajectories of the particle plume for all the one-port powder injection conditions. As shown in Figure 5.2, within the distance from the centerline of the internal injection port to the centerline of the straight external injection port (21mm), the particles injected through internal injection port would travel along a mean trajectory where the plasma jet temperature is in the range of around 8,000 to 11,000K (Figure 5.3 a) and the plasma jet velocity in the range of 900 to 1100 m/s (Figure 5.3 b). This region is the core region of the plasma jet where the particles would experience the most efficient heating and acceleration. However, for the externally injected particles, they only travel along the periphery of the core region of the plasma jet, and the plasma jet temperature and velocity along their mean trajectory would be much lower. For the particles injected through the straight external injection port, the maximum heating temperature by the plasma jet they experience is only around 4,500K (level 7 in Figure 5.3 a). Tilting the injection angle towards the counter-stream direction would make the particles penetrate into a hotter region of the plasma, for example level 10 of around 7,400K in Figure 5.3a. However, their residence time inside the hotter region is also very short. Thus, from the simulation results, it could lead to a general conclusion that particle injected through the internal

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injection port would have the highest mean velocity and temperature. The particles injected through the counter-flow tilting external injection port would have a higher particle mean velocity and temperature than those injected through the straight external injection port.

5.3 Radial Distributions of Mean Particle Velocity and Diameter

5.3.1 Internal Injection of Powders

Figures 5.4-5.7 show the radial distribution of mean particle diameter and velocity of YSZ powders injected through one internal transverse injector subjected to the spraying conditions listed in Table 5-2. The respective influence of the plasma gas flow rate, arc current level, carrier gas flow rate and powder feed rate on the particle diameter and velocity distributions was individually characterized by keeping other parameters constant while changing the parameter of interest. Particles were injected towards the positive direction of the x -axis with the axis $x = 0$ corresponding to the centerline of the plasma torch.

Table 5-2 List of plasma spraying parameters for internal injection of powders through one transversal injector.

Cases	Plasma Gas		Arc Current <i>Ampere</i>	Carrier Gas		Powder Feed Rate	
	<i>psi</i>	<i>slm</i>		<i>psi</i>	<i>slm</i>	<i>rpm</i>	<i>g/min</i>
I 1	40	41.47	850	40	5.22	2.2	20
I 2	60	56.85	850	40	5.22	2.2	20
I 3	80	72.23	850	40	5.22	2.2	20
I 4	60	56.85	500	40	5.22	2.2	20
I 5	60	56.85	700	40	5.22	2.2	20
I 6	60	56.85	900	40	5.22	2.2	20
I 7	60	56.85	850	20	3.16	2.2	20
I 8	60	56.85	850	60	7.28	2.2	20
I 9	60	56.85	850	40	5.22	1.1	10
I 10	60	56.85	850	40	5.22	3.3	30

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It was well known that the transverse injection of particles comprised of different sizes would lead to particle segregation inside the plasma jet. Larger particles penetrated further into the plasma jet due to their larger inertia while smaller particles tended to concentrate on the same side of the injector. However, it should be pointed out that at every measured point there was a spectrum of different particle sizes. The complex collisions between particles and particle-injector wall would lead to changes of in-flight particle trajectories. Thus, smaller particles could appear on the right-hand side (opposite to the powder injector) and there were also larger particles on the left-hand side (same side of the powder injector), as shown previously in **Figure 4.11 (a)** in **Chapter 4**. The mean particle diameter only represented the dominant size group at a particular location. The particle segregation according to their diameter inside the plasma jet was confirmed in the present experiments. The measurements of the mean particle diameter distribution carried out for all the one-port transverse injection cases showed that the mean particle diameter increased gradually towards the powder injection direction, i.e. the positive direction of x -axis. The exception was, for some cases such as **Case II** and **I2**, the increment stopped after the mean particle diameters reached their peak values. After that, it started to drop further towards the positive x -axis direction. This phenomenon was actually related to the plasma jet behavior and the particle temperature history along their in-flight trajectories. Plasma jet was characterized by strong turbulent mixing with the surrounding atmosphere, which entrained ambient air into the plasma. The amount of entrained air increased with increasing distance from the nozzle exit. On one hand, air molecules would be dissociated at a temperature lower than the ionization temperature of argon, and the dissociation of the entrained air cooled the plasma jet efficiently due to high energy consumption. On the other hand, air had higher specific heat capacity than argon gas, which meant that air needed more energy than argon to reach the same temperature. When more surrounding air

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was entrained into argon gas, temperature within the plasma jet would subsequently be reduced. Due to the higher mass fraction of air near the fringe of the plasma jet, plasma temperature there would be much lower than that in the core region, and even lower than the melting point of YSZ particles. Larger particles were expected to cross the plasma jet and reach the fringe rapidly. During the short residence time inside the hot core of plasma jet, larger particles could at most have their surface melted, and some of them might even not melted at all. For larger particles partially melted on their surface, they cooled down and started to solidify shortly after leaving the core region of the plasma jet. The solidification of larger particles was especially prominent when they reached the low temperature fringe region. During the process of particle solidification, the crystallization, starting from the particle surfaces, tended to form dendritic structures, and the surface turned from smooth to rough. For the PDA measurement, either the solidification or unmelting of these larger particles would lead to weak and noisy signals since the incident laser light were scattered arbitrarily from the particle rough surface and consequently, resulted in lower signal-to-noise ratio and rejection of particles. The farther the measurement point was from the centerline of the plasma jet, the higher possibility for larger particles to be rejected. Thus, mean particle diameter showed a decreasing trend after reaching a peak value along the powder injection direction.

Based on the knowledge of particle size distribution inside the plasma jet, it could be seen that smaller particles appearing near the centerline acquired higher velocity. In plasma spraying, the main contribution for the acceleration of in-flight particles was the drag force exerted by the plasma, which was in a function of particle size and relative velocity between plasma and particle velocity. The equation for the axial motion of a spherical particle of mass m_p (kg) had the form:

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$$m_p \frac{du}{dt} = F_D \quad (5-1)$$

u was the particle velocity (m/s), and F_D was the drag force (Newton) which was defined by,

$$F_D = \frac{1}{2} C_D \rho_g A_p (U - u)^2 \quad (5-2)$$

where C_D was the drag coefficient, ρ_g were the plasma density (kg/m^3), A_p the surface area of the particle (mm^2) and U was the plasma velocity (m/s). The simplest formula for C_D as function of the particle Reynolds number Re was given by,

$$C_D = \frac{24}{\text{Re}} \quad (5-3)$$

$$\text{Re} = \frac{\rho_g d_p (U - u)}{\mu_g} \quad (5-4)$$

where d_p was particle diameter (m) and μ_g was the dynamic viscosity of plasma (kg/m/s).

Thus, the acceleration of particle along the axial direction could be derived by substituting **Equations 5-2 to 5-4** into **Equation 5-1** and after re-arrangement, expressed as,

$$\frac{du}{dt} = \frac{18\mu_g}{\rho_p d_p^2} (U - u) \quad (5-5)$$

where ρ_p is the particle density (kg/m^3). From **Equation 5-5** it was obvious that for powders of the same material in a given plasma jet, the acceleration of particle was inversely proportional to the square of the particle diameter and proportional to the velocity difference between the plasma and particle. In another word, smaller particles were much easier to accelerate, and the acceleration would be greatly enhanced when they traveled along a trajectory around the centerline of the plasma jet, where the plasma velocity was much higher than particle velocity. The mean particle velocities obtained the highest value at around the centerline of the plasma torch, where smaller particles

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populated and plasma velocity was also higher, and decreased with the distance from the centerline. At the right-hand fringe of the particle plume, small particles populated due to their insufficient momentum to penetrate inside the plasma jet and therefore remained at the periphery of the plasma jet. Particles there would not experience sufficient acceleration and thus the mean particle velocity was lower at that location. At the left-hand fringe, where the mean particle velocity was also low, larger particles were concentrated after they crossed the plasma jet with a short residence time. The mean particle velocities exhibited a skewed Gaussian distribution about the plasma jet axis (the centerline of the plasma torch), slanting more towards the positive direction of the x -axis.

As expected, increasing the plasma gas flow rate and arc current level brought pronounced increase to the particle in-flight velocity due to the increased plasma jet velocity (**Figure 5.4 and 5.5**). However, it was interesting to note that the mean particle diameter distribution varied significantly under different plasma gas flow rate, but it did not vary too much with arc current level. This phenomenon might be attributed to the particle penetration mechanism with respect to the plasma jet of different momentum. Increasing the plasma gas flow rate increased the plasma jet density as well as its velocity, which made the smaller particles, whose momentum was much lower, more difficult to penetrate into the plasma jet and much more likely to be bounced away. Larger particles also met greater resistance when entering the plasma jet, and could not penetrate faraway from the plasma jet axis. Thus, the entire particle plume dispersion would be narrower with higher plasma gas flow rate. At the locations near the plasma jet axis, with the decreased number of smaller particles and increased number of larger particles, the PDA measured average particle diameter would increase with the plasma gas flow rate. On the other hand, when increasing the arc current level only without altering other parameters, the plasma jet

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velocity would be expected to increase significantly. However, the plasma jet density would not be of much difference due to the same plasma gas flow rate and thus the almost same plasma density. The similarity of the mean particle diameter distributions under different arc current levels might lead to the conclusion that the particle penetration inside the plasma jet was predominantly influenced by the plasma jet density, and the increase of the plasma jet velocity only had minor contribution.

Increasing the carrier gas flow rate lead to the most prominent change in the particle dispersion inside the plasma jet, as shown in **Figure 5.6**. The entire particle plume was shifted towards the powder injection direction, as reflected by the distribution curves of the mean particle velocity and diameter. The maximum value of the mean particle velocity did not change much with the increase in carrier gas flow rate, and the distributions maintained the similar trend. The mean particle diameter distributions, however, exhibited obvious difference under the lower and higher carrier gas flow rate conditions. When the carrier gas flow rate was lower, the particle dispersion inside the plasma jet would be confined in a small region. A large portion of the particles was populated near the plasma jet axis without penetrating faraway. Increasing the carrier gas flow rate made the particle segregation phenomenon more pronounced, with only the larger particles having enough momentum to cross the plasma jet. It could be observed that the maximum mean particle diameter in the lower carrier gas flow rate conditions was obviously lower than that in the higher carrier gas flow rate conditions. The reason was due to the fact that less particles were segregated under lower carrier gas flow rate, which made the averaged particle diameter lower. On the other hand, the particle evaporation phenomenon was believed to have taken place and played an important role. For particles with very low thermal conductivity, such as YSZ powders used in the present experiments, the surface of larger

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particles might start to evaporate while their cores were still solid during their flight inside the plasma jet. And the evaporation was especially biased towards smaller particles because they could be heated up more easily and rapidly. As the particle surface temperature approached the boiling point (about 5000K), the vaporization rate would become very high, and the vaporized mass fraction could be higher than 20%. Therefore, the in-flight mean particle diameter detected by PDA at every measuring location was actually determined simultaneously by evaporation and penetration of particles into the plasma jet. Based on the particle segregation according to their inertia, evaporation caused larger particles a decrease in their diameter. Smaller particles might be partially or totally evaporated, depending on their size and trajectories inside the plasma jet. Even for some partially evaporated smaller particles, they might not be detected by PDA for the reason that the laser light intensities scattered would be too weak to reach the threshold signal needed for the PDA detection. As for the particles injected inside the plasma jet with 3.16slm (**Case I7** in **Table 5-2**) of carrier gas flow rate, most of them were concentrated near the plasma jet axis and were believed to have traveled inside the core region of the plasma jet. Under this condition, many of them might be overheated and surface-evaporated, which resulted in decrease of their diameters. The evaporation of particles would diminish with the increased particle penetration distance from the plasma jet axis for larger particles when increasing the carrier gas flow rate, where cooling effect to the plasma jet might also have contributed to the reduced particle overheating.

Increasing the powder feeder rate from 10g/min to 30g/min did not change the mean particle velocity and diameter significantly. Nevertheless, a small decrease of the mean particle velocity could be observed with the increase of powder feed rate, as shown in **Figure 5.7**. This could be readily explained by the fact that for a given plasma jet, the

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momentum transferred from the plasma jet to the individual particle would decrease with the increased number of in-flight particles.

As shown above in **Figure 5.4-5.7**, the skewed particle velocity distribution and the aerodynamic classification of particle size produced by the transverse injection of particles through one injection port might not be favorable for forming an uniform coating. Larger particles with lower velocities tended to cause porosity and insufficient adhesion between individual splats. The large velocity gradients may result in a heterogeneous density distribution of the coating. Additionally, since the particle plume always tilted towards the opposite side of the injection port, particles were accelerated and heated only within part of the plasma jet, which caused insufficient utilization of the heat source. Compared with conventional one-port injection, the mean particle velocity and diameter of the powders injected from the opposite two injectors corresponding to the operation conditions listed in **Table 5-3** exhibited axi-symmetric radial distributions, as shown from **Figures 5.8-5.11**. The particle plume was almost symmetrically distributed about the centerline of the plasma torch, and the edges of the particle plume were much nearer to the centerline. Under these circumstances, the number of particles moving along the periphery of the plasma jet decreased. The distribution curves of particle characteristics for two-port injection were far from a simple linear superposition of two individual distribution curves of particles injected from each of the opposite injectors. Due to the interaction of the opposite two powder-gas flows, particles injected from one injector met the opposing retarding force applied by the mixture flow from the opposite injector when trying to cross the plasma jet. Accordingly, particle trajectories were changed in that particles tended to travel along the paths closer to the centerline of the plasma jet. It could be expected that some particles whose in-flight trajectories happened to be along the centerline after the interaction

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between the two mixture flows spent the longest time in the plasma jet and acquired higher velocity and temperature. This provided the explanation that the maximum mean particle velocity always appeared around the plasma jet axis. In addition, unlike the obvious segregation of particles according to their size in the case of one-port injection (Figure 5.4-5.7), injection of powders from two opposite ports resulted in axi-symmetrical distribution of mean particle diameter. On either side of the plasma jet axis, there were larger particles that crossed the plasma jet from the opposite side injector, together with smaller ones injected from the same side injector that penetrated inside the plasma jet but could not travel beyond the centerline. Thus, the averaged particle diameter also exhibited an almost axi-symmetric distribution.

Table 5-3 List of plasma spraying parameters for internal injection of powders through opposite two transversal injectors.

Cases	Plasma Gas		Arc Current <i>Ampere</i>	Carrier Gas		Powder Feed Rate	
	<i>psi</i>	<i>slm</i>		<i>psi</i>	<i>slm</i>	<i>rpm</i>	<i>g/min</i>
II 1	40	41.47	850	30	4.19	2.2	20
II 2	60	56.85	850	30	4.19	2.2	20
II 3	80	72.23	850	30	4.19	2.2	20
II 4	60	56.85	500	30	4.19	2.2	20
II 5	60	56.85	700	30	4.19	2.2	20
II 6	60	56.85	900	30	4.19	2.2	20
II 7	60	56.85	850	10	2.13	2.2	20
II 8	60	56.85	850	50	6.25	2.2	20
II 9	60	56.85	850	30	4.19	1.1	10
II 10	60	56.85	850	30	4.19	3.3	30

In spite of the prominent difference in the radial distributions of the mean particle velocity and diameter between the one-port and two-port powder injection schemes, the influence of the spraying parameters on the variations of the in-flight particle characteristics for the two-port injected particles follows the similar trend as those of one-port injected particles.

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Increasing the plasma gas flow rate and arc current level increased the particle velocity significantly, as shown in **Figure 5.8** and **5.9**. When the plasma gas flow rate was lower, both the plasma jet density and its velocity were lower. In this case, larger particles injected from each of two injectors were much easier to cross the plasma jet and populated at the opposite side of their respective injector faraway from the plasma jet axis. Many smaller particles would be able to enter the plasma jet, and they were mostly concentrated around the plasma jet axis. Thus, the averaged particle diameter would be smaller at around the plasma jet axis locations while larger at the two edge locations of the particle plume under the lower plasma gas flow rate condition. Increasing the plasma gas flow rate made the smaller particles more difficult to penetrate inside the plasma jet while the larger particles populate nearer to the plasma jet axis. Consequently, the averaged particle diameters at around the plasma jet axis would increase and those at the edges of the particle plume would decrease with increasing the plasma gas flow rate. Similarly with the one-port injection scheme, increasing the arc current level increased the mean particle velocities, but had no significant influence on the mean particle diameter distribution due to the almost constant plasma jet density.

Increasing carrier gas flow enlarged the transverse extent of the particle plume, which also led to a drop in mean particle velocities at the edges, as shown in **Figure 5.10**. However, the maximum value of mean particle velocity in the centerline increased correspondingly. As mentioned earlier, higher carrier gas flow rates corresponded to higher particle injection momentum, which altered particle trajectory earlier at a position closer to the nozzle exit. Thus, particles traveling along the centerline remained in the plasma jet for a longer time and thus acquired a higher velocity. It should be emphasized here that for the two-port powder injection scheme, the powder feed rate and the carrier gas flow rate applied to each

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of the two injectors were approximately only half of those respective two settings indicated on the parameter control board of the plasma spray system. When the carrier gas flow rate was set as 2.13slm in the control board, particles injected through each of the two injectors were actually applied with around 1.06slm carrier gas flow rate. In this circumstance, the carrier gas flow rate regarding to the plasma jet momentum would be too low, and it would most probably that only the larger particles could penetrate inside the plasma jet while smaller ones would mostly accumulate along the periphery of the plasma jet without penetrating inside. When the carrier gas flow rate increased, larger particles crossed the centerline of the plasma torch and spread further towards the opposite direction of their injection port, while many smaller particles would be able to penetrate inside the plasma jet. Therefore, the increase of the carrier gas flow rate resulted in two large particle concentration areas appearing on either side of plasma jet axis, while the mean particle diameter became smaller at around the centerline of the plasma torch.

Increasing the powder feed rate from 10g/min to 30g/min brought no significant variations to curves of the mean particle velocity and diameter, except that the particle mean velocity was higher under lower powder feed rate condition, which was similar to the trend for the one-port injection of powders (**Figure 5.11**).

5.3.2 External Injection of Powders

As mentioned earlier, for a given plasma jet, the particle injection momentum has to be optimized with that of the plasma jet so that the particles would be able to travel along the trajectories to be melted effectively. It should be emphasized that the most important interaction region between the plasma jet and the particles would be in the vicinity of the powder injector exit, where the change of the particle traveling path determines the

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consequent particle in-flight trajectories inside the plasma jet. As previously illustrated in **Figure 5.3**, the plasma jet temperature and velocity are the highest inside the anode constriction region, i.e. inside the plasma torch, and the plasma density there is also much higher than that in the outside region of the plasma torch. Thus, it can be expected that under the same carrier gas flow rate condition, the particles injected through the internal injector would experience more resistant force to penetrate inside the plasma jet than those injected through external injector. In another word, external injection of powders would enable the injected particles to penetrate into the plasma jet easier due to a lower momentum of the expanding plasma jet. In addition, particles injected through the internal injector would reside in the hottest region of the plasma jet for a longer time, and it could be expected that the particle velocity and temperature, and thus, the coating deposition efficiency, would be higher [20, 67].

In this section, the radial distribution for the mean particle diameters and velocities under different spraying conditions for external injection of powders were measured. For each external powder injection scheme as schematically shown in **Figure 5.1 c-f**, the influences of the plasma gas flow rate and carrier gas flow rate were investigated. **Table 5-4** and **5-5** listed the spraying conditions for the external injection of powders for transversal injection and 75° counterflow injection respectively.

Figure 5.12-15 show the radial distributions of mean particle diameter and velocity for the powders injected externally through one and opposite two straight injectors under different plasma gas flow rate and carrier gas flow rate conditions. Comparing with the corresponding internal powder injection conditions (**Figure 5.4, 5.6, and Figure 5.8, 5.10**), it could be observed that the maximum mean particle velocity dropped by 14%-25% and

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the maximum mean particle diameter dropped by 20%-30%. The decrease in the particle velocity could be attributed to the shorter acceleration time experienced by the externally injected powders, and at the same time the overall lower plasma velocities along the traveling path of those powders, as mentioned above. Similarly, the externally injected powders traveled along the peripheral of the core region of the plasma jet, resulting in a shorter heating time. In this case, only the smaller particles would be able to be melted, and most of the larger particles remained in the unmelted state and would most probably be rejected by the PDA measurement. Therefore, the mean particle diameter would decrease. For external injection of powders through one straight injector, increasing the plasma gas flow rate would lead to overall increase in the particle velocities and diameters, as shown in **Figure 5.12**, which was similar to the one port internal injection condition (**Figure 5.4**). Increasing the carrier gas flow rate would make the velocity distribution curve shift towards the powder injection direction, as shown in **Figure 5.13**. The particle velocity was decreased under higher carrier gas flow rate due to the rapid transit of the particles through the plasma jet. For particles injected externally via two straight injectors opposite to each other, increasing the plasma gas flow rate also led to an increase in the maximum particle mean velocity and diameter, which occurred around the centerline of the plasma jet, as shown in **Figure 5.14**. Increasing the carrier gas flow rate resulted in increase of the maximum mean particle velocity but decrease in the particle diameter (**Figure 5.15**), which was also similar with the internal injection condition and the reason was the same (**Figure 5.10**).

Tilting the powder injector towards the counterflow direction increased the powder residence time inside the plasma jet, which resulted in a little higher particle mean velocities and diameters, as shown in **Figure 5.16-19**. However, under this circumstance,

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particles would also be much easier to cross the plasma jet under lower plasma gas flow rate or higher carrier gas flow rate conditions, which made the mean particle velocity and diameter decrease, as shown in case *ET1* and *ET5*.

Table 5-4 List of plasma spraying parameters for external injection of powders through straight injector(s) (zero degree injection).

Cases	Plasma Gas		Arc Current	Carrier Gas		Powder Feed Rate	
	<i>psi</i>	<i>slm</i>		<i>psi</i>	<i>slm</i>	<i>rpm</i>	<i>g/min</i>
One-Port	ES 1	40	41.47	40	5.22		
	ES 2	60	56.85	40	5.22		
	ES 3	80	72.23	40	5.22	2.2	20
	ES 4	60	56.85	20	3.16		
	ES 5	60	56.85	60	7.28		
Two Port	ES 6	40	41.47	30	4.19		
	ES 7	60	56.85	30	4.19		
	ES 8	80	72.23	30	4.19	2.2	20
	ES 9	60	56.85	10	2.13		
	ES10	60	56.85	50	6.25		

Table 5-5 List of plasma spraying parameters for external injection of powders through tilting injector(s) (-75 degree counter-flow injection).

Cases	Plasma Gas		Arc Current	Carrier Gas		Powder Feed Rate	
	<i>psi</i>	<i>slm</i>		<i>psi</i>	<i>slm</i>	<i>rpm</i>	<i>g/min</i>
One Port	ET 1	40	41.47	40	5.22		
	ET 2	60	56.85	40	5.22		
	ET 3	80	72.23	40	5.22	2.2	20
	ET 4	60	56.85	20	3.16		
	ET 5	60	56.85	60	7.28		
Two-Port	ET 6	40	41.47	30	4.19		
	ET 7	60	56.85	30	4.19		
	ET 8	80	72.23	30	4.19	2.2	20
	ET 9	60	56.85	10	2.13		
	ET10	60	56.85	50	6.25		

5.4 Radial Distributions of Particle Concentration and Volume Flux

As mentioned in the previous chapter, particle concentration and volume flux measured by the PDA could be used to estimate the amount of molten particles finally deposited on the substrate. **Figures 5.20-35** show the radial distributions of the particle concentration and volume flux under the various spraying conditions investigated previously (**Table 5-2 – Table 5-5**) For most of the cases, it could be noticed that the PDA measured particle concentration distribution followed almost the same variation trend as the particle volume flux distribution with the spraying parameters. This phenomenon might be attributed to the reason that for either internal injection or external injection of powder conditions, the variation of the number of the measured particles with spraying parameters played a more important role than the variation of particle diameters on influencing the particle volume flux. Furthermore, both the PDA measured particle concentration and volume flux were quite sensitive to the spraying parameters. For example, in the case of internal injection of powders through one injection port, increasing the plasma gas flow rate from 41.47slm to 72.23slm brought a 64% decrease of the PDA measured particle concentration and a 41% decrease of the particle volume flux for the peak values on the respective curve, as shown in **Figure 5.20 (a) and (b)**. The integrated value over the particle concentration or volume flux distribution along the radial direction would be able to provide comparable results among all the spraying conditions investigated, from which the quantitative evaluation of the optimal plasma spraying parameter setting could be obtained. **Table 5-6** was a matrix containing the integrated particle volume flux values along the radial direction for each of the distribution curves corresponding to the spraying conditions under investigation. The integrated values were all dimensionless with the highest value, which was for the case of internal injection of powder through two opposite injectors II10. From this matrix, it could be observed, also as expected, that the integrated volume flux values of the particles

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injected through external injection port were significantly lower than those for internal injection of particles. It was normal since the particles being injected through external injectors would penetrate inside the plasma jet only at the tail of the plasma jet core region, which lead to insufficient heating of the powders and a decreased number of molten particles. Under this condition, most probably only the smaller particles would be able to be melted due to their smaller heat capacity, which could also be demonstrated by the smaller mean particle diameters as mentioned before. Tilting the powder injector towards the counter-flow direction resulted in the increased particle residence time inside the core region of the plasma jet, thus led to a slightly increase of the PDA measured particle volume flux. By comparing the integrated particle volume flux under the same spraying conditions for one-port and two-port injection conditions, it could be noticed that in most cases the latter was higher than the former. This phenomenon could be readily explained by the fact that, under the same powder feed rate, the amount of particles injected from each of the two opposite injectors was approximately half of that for one-port injection. For two-port injection, more particles were traveling along the trajectories nearer to the core region of the plasma jet and therefore the heat transfer process occurred efficiently from the plasma to particles. The combined effect of the decreased local particle loadings in the plasma jet and more efficient heating of particles for two-port injection of powders lead to an increased number of melted particles. In this case, the particle sphericity and surface smoothness would be improved and consequently resulted in an overall higher particle volume flux.

The variation of the PDA measured particle volume flux with the spraying conditions could be easily and quantitatively characterized, which would make the determination of the optimal spraying conditions more efficient and reliable. In order to demonstrate the

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validity of using the PDA measured particle volume flux to determine the optimal spraying conditions, the PDA measured particle volume flux would be compared with the coating microstructure and deposition efficiency produced under the same spraying conditions in the next chapter.

Table 5-6 The dimensionless integrated value of the PDA measured particle volume flux along the radial direction for all the plasma spraying conditions investigated in the present chapter.

		One Port		Two Port	
Internal Injection (Straight Injector)		I1	0.712486	II1	0.782739
		I2	0.473979	II2	0.64208
		I3	0.391051	II3	0.488414
		I4	0.229359	II4	0.315337
		I5	0.377497	II5	0.610708
		I6	0.565114	II6	0.756165
		I7	0.797129	II7	0.509345
		I8	0.398666	II8	0.637556
		I9	0.267732	II9	0.424116
		I10	0.751028	II10	1.0
External Injection	Straight Injector	ES1	0.146756	ES6	0.282158
		ES2	0.206434	ES7	0.303614
		ES3	0.258653	ES8	0.274806
		ES4	0.296986	ES9	0.252197
		ES5	0.112274	ES10	0.296741
	Tilting Injector	ET1	0.19375	ET6	0.388065
		ET2	0.23276	ET7	0.347364
		ET3	0.283433	ET8	0.292949
		ET4	0.423464	ET9	0.298454
		ET5	0.13465	ET10	0.365067

5.5 Summary

With the aid of the PDA technique, the in-flight particle characteristics of the atmospherically plasma sprayed powders were systematically investigated with respect to some typical spraying parameters, i.e. the powder injection scheme, the plasma gas flow rate, carrier gas flow rate, arc current level and powder feed rate. Particle in-flight velocity,

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diameter, concentration and volume flux distributions were investigated due to their importance in providing the information of the momentum and heat transfer from the plasma jet to the particles and in further predicting the coating properties. The commercially available Ytria (8wt.%) partially stabilized Zirconia (YSZ) powders were sprayed through different arrangements of powder injectors, and the PDA system was chosen carefully to achieve the highest measured data validation rate.

Results were presented at a fixed standoff distance of 80 mm from the exit of the plasma torch nozzle, along the radial direction in the center of the plasma jet. Regarding to each dominating spraying parameter under investigation, the particle in-flight behaviors were characterized only by varying that parameter while keeping other parameters constant. By doing so, a clear understanding of the variation trend of the particle characteristics with the parameter of interest would be obtained. Although the importance of investigating the particle in-flight behavior in determining the optimal setting of the spraying parameters has been addressed before, none of the previous researches could be able to provide a direct and quantitative criterion. In the present experiments, with the optimized PDA measurements, the integrated values of the PDA measured particle volume flux along the radial direction for the individual setting of the spraying parameters were adopted as the evaluation criterion to determine the optimal plasma spraying setting, from which it could be supposed that the powders injected inside the plasma jet through two opposite internal injectors would be able to experience more efficient heating and thus more powders could be melted inside the plasma jet. In addition, from the radial distribution curve of the particle concentration and volume flux, it could also be expected that the coatings formed by the powder of two-port injection would be more uniform with flat surface.

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Figure 5.1 Schematically illustration of the six powder injection schemes investigated; (a) internal one straight port injection, (b) internal two opposite straight ports injection, (c) external one straight port injection, (d) external two opposite straight ports injection, (e) external one tilting port injection, and (f) external two opposite tilting ports injection.

Figure 5.2 Illustration of the position and dimension of the different powder injectors relative to the plasma torch.

Figure 5.3 Numerical modeling of the (a) iso-temperature distribution and (b) iso-velocity distribution of the plasma jet produced under spraying conditions listed in Table 5-1.

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Figure 5.4 The radial distributions of mean particle velocity and diameter under different plasma gas flow rate conditions for internal injection of powder through one straight port.

Figure 5.5 The radial distributions of mean particle velocity and diameter under different arc current conditions for internal injection of powder through one straight port.

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Figure 5.6 The radial distributions of mean particle velocity and diameter under different carrier gas flow rate conditions for internal injection of powder through one straight port.

Figure 5.7 The radial distributions of mean particle velocity and diameter under different powder feed rate conditions for internal injection of powder through one straight port.

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Figure 5.8 The radial distributions of mean particle velocity and diameter under different plasma gas flow rate conditions for internal injection of powder through opposite two straight ports.

Figure 5.9 The radial distributions of mean particle velocity and diameter under different arc current conditions for internal injection of powder through opposite two straight ports.

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Figure 5.10 The radial distributions of mean particle velocity and diameter under different carrier gas flow rate conditions for internal injection of powder through opposite two straight ports.

Figure 5.11 The radial distributions of mean particle velocity and diameter under different powder feed rate conditions for internal injection of powder through opposite two straight ports.

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Figure 5.12 The radial distributions of mean particle velocity and diameter under different plasma gas flow rate conditions for external injection of powder through one straight port.

Figure 5.13 The radial distributions of mean particle velocity and diameter under different carrier gas flow rate conditions for external injection of powder through one straight port.

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Figure 5.14 The radial distributions of mean particle velocity and diameter under different plasma gas flow rate conditions for external injection of powder through opposite two straight ports.

Figure 5.15 The radial distributions of mean particle velocity and diameter under different carrier gas flow rate conditions for external injection of powder through opposite two straight ports.

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Figure 5.16 The radial distributions of mean particle velocity and diameter under different plasma gas flow rate conditions for external injection of powder through one tilting port.

Figure 5.17 The radial distributions of mean particle velocity and diameter under different carrier gas flow rate conditions for external injection of powder through one tilting port.

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Figure 5.18 The radial distributions of mean particle velocity and diameter under different plasma gas flow rate conditions for external injection of powder through opposite two tilting ports.

Figure 5.19 The radial distributions of mean particle velocity and diameter under different carrier gas flow rate conditions for external injection of powder through opposite two tilting ports.

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Figure 5.20 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different plasma gas flow rate conditions for internal injection of powder through one straight port.

Figure 5.21 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different arc current conditions for internal injection of powder through one straight port.

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Figure 5.22 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different carrier gas flow rate conditions for internal injection of powder through one straight port.

Figure 5.23 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different powder feed rate conditions for internal injection of powder through one straight port.

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Figure 5.24 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different plasma gas flow rate conditions for internal injection of powder through opposite two straight ports.

Figure 5.25 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different arc current conditions for internal injection of powder through opposite two straight ports.

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Figure 5.26 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different carrier gas flow rate conditions for internal injection of powder through opposite two straight ports.

Figure 5.27 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different powder feed rate conditions for internal injection of powder through opposite two straight ports.

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Figure 5.28 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different plasma gas flow rate conditions for external injection of powder through one straight port.

Figure 5.29 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different carrier gas flow rate conditions for external injection of powder through one straight port.

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Figure 5.30 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different plasma gas flow rate conditions for external injection of powder through opposite two straight ports.

Figure 5.31 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different carrier gas flow rate conditions for external injection of powder through opposite two straight ports.

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Figure 5.32 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different plasma gas flow rate conditions for external injection of powder through one tilting port.

Figure 5.33 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different carrier gas flow rate conditions for external injection of powder through one tilting port.

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Figure 5.34 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different plasma gas flow rate conditions for external injection of powder through opposite two tilting ports.

Figure 5.35 The PDA measured radial distribution of (a) particle concentration and (b) particle volume flux under different carrier gas flow rate conditions for external injection of powder through opposite two tilting ports.

Chapter 6

Investigation on the Coating Microstructure and Deposition Efficiency

6.1 Microstructure of the Plasma-Sprayed Coatings

The ultimate purpose of investigating the influence of the plasma spray parameters on the particle in-flight characteristics was to predict the plasma sprayed coating properties and to determine the optimal parameter settings to achieve the desired coatings. It has been well known that many of the coating properties were closely related to the coating microstructure, which was again primarily determined by the impacting particle velocity, diameter and temperature distributions. Theoretically, coatings formed by the successive building-up of fully melted particles impacting orthogonally to the substrate with high velocities would achieve the optimal mechanical and thermal properties. Such coatings would be much denser and tougher, with higher adhesion/cohesion strength. However, due to the large velocity and temperature gradients of the plasma jet, particles with different initial diameters and injection velocities would experience quite different accelerations and heating treatments inside the plasma jet. Some particles would not be able to penetrate inside the core region of the plasma jet or stay there long enough to be melted. These unmelted particles would most probably not be able to adhere onto the substrate and would bounce off upon impacting on the substrate. Some particles would only be surface-melted, most of which would be expected to be larger particles. These particles might be imbedded onto the coatings, and their existence would modify the coating microstructure and properties. In conclusion, the plasma sprayed coatings had a structure totally different from

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that of the corresponding bulk materials, and it was typically characterized by layered structure formed by the molten particles and the grains of entrapped unmelted particles, intermediate with various sizes of porosities.

In the previous literature, most of the research contributed to investigating the influence of the individual spraying parameters on the in-flight particle parameters or on the coating properties separately without coupling them together to make a comparison. By doing so, although the respective variation trend of the in-flight particle parameters and the coating properties with the spraying parameters could be roughly understood, how the in-flight particle parameter change would influence the coating properties and what were the optimal settings of the spraying parameters for producing the desired coatings remained vague. In this section, coatings were sprayed for the microstructure analysis under the operation conditions corresponding to some typical cases adopted for the PDA investigations as demonstrated in the previous chapter. Although the in-flight particle temperature were not measured in the present experiments, it could be reasonably inferred from the comparison between the in-flight particle velocity and diameter measured by the PDA introduced in **Chapter 5** and the microstructure of the coatings produced under the same spraying conditions.

6.2 Preparation of the Plasma-Sprayed Coating and Single Splat

6.2.1 Spraying Strips for Coating Microstructure Analysis

Coatings were sprayed on the 70mm×50mm×2mm thick mild steel substrates without pre-heating of the substrates. All the substrates were almost identical in dimension, with two holes of 4mm in diameter drilled at each side for the substrate to be fastened on the substrate holder. The substrates were sandblasted and air cleaned to have a surface roughness of 5 to 7 μm . The orientation of the substrate with respect to the plasma torch was shown previously in **Figure 4.16**. Each substrate was placed at the standoff distance of 80mm from the nozzle exit perpendicularly to obtain a comparable result with the previous PDA measurements. The plasma torch was mounted on the computer-controlled 3-D traversing rig mentioned earlier, and was traversed horizontally and vertically to produce a flat top surface of the coating. All the coatings were then cut by a diamond saw perpendicularly to the traverse movement direction of the plasma torch so that the cross sectional plane of the coatings could be observed. Each specimen, mounted inside the thermoplastic matrix with its cross sectional plane exposing to the air, as shown in **Figure 6.1**, was consequently grinded and polished with Struer's Abramin grind/polish system and brought under Scanning Electron Microscopy (SEM) for microstructure examination. The spraying conditions for producing the plasma sprayed coatings were listed in **Table 6-1**.

6.2.2 Single Splat Capture

The morphology of single splat formed by individual particles impacting on a smooth substrate was also investigated. In this case, the substrates were much smaller (15mm×10mm×2mm) than those used for coating deposition in order to facilitate the process of polishing of the substrate surface to achieve an almost mirror finishing. The substrates were submerged into ethanol and cleaned with ultrasonic cleaner and air-dried

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after polishing. After that, the substrate was fixed on the substrate holder which was placed at the standoff distance of 80mm from the exit of the torch nozzle. The substrate holder could slide with an adjustable speed. With a very low powder-feeding rate and the spraying reaching the steady state, the substrate moved rapidly through the plasma jet once to capture a number of single splats on the substrate with minimum overlapping. The splats scattered throughout the substrate and Scanning Electron Microscopy (SEM) was then used to observe their morphologies.

6.3 Coating Microstructure Examination

As mentioned earlier, the plasma sprayed coating is formed by the accumulation of the sprayed particles. Thus, the individual morphology of these sprayed particles upon impacting on the substrate would determine the overall coating microstructure and properties. Depending on their respective residence time inside the core region of the plasma jet and their physical properties, the plasma sprayed particles might be completely melted, partially melted, or even totally unmelted but still sticking on the substrate. **Figure 6.2** and **6.3** show the various morphologies of the single splat formed by the impacting spherical agglomerated YSZ particles used in the present experiments. As introduced in **Chapter 4**, the spherical agglomerated YSZ powders were fabricated by spray drying of the much smaller grains with the diameters around $1\ \mu\text{m}$. Upon exposing to the plasma jet, particles being fully melted would exhibit a compact disc-shape when impacting on substrate or the pre-sprayed layer, as shown in **Figure 6.2 (a)**. For some smaller particles that were completely melted inside the plasma jet, they might have already cooled down and re-solidified upon impacting on the substrate due to their lower heat capacity, as shown in **Figure 6.2 (b)**. Particles only being surface-melted might also be included in the coating, but their inner part still remained as solid agglomeration of the smaller grains, as shown in **Figure 6.3**.

Figures 6.4 to 6.8 and **Figures 6.9 to 6.13** show the microstructures of the coatings sprayed respectively with one-port and two-port powder injection scheme under the spraying conditions listed in **Table 6-1**. The influence of the individual spraying parameters on the coating microstructure could be readily observed by comparing the microstructure images of coatings produced under different values of the parameter of interest while keeping other parameters constant. As a general view of all the images, it

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could be observed that the coating microstructures were characterized by some large scale cavities or cracks together with many small scale porosities. It should be pointed out that these large scale cavities or cracks were actually caused by the detachments of the unmelted particles imbedded in the coatings during the process of grinding and polishing of the coating cross-sectional plane for the SEM observation. As shown above in **Figure 6.2** and **6.3**, the existence of the partially melted particles decreased the local mechanical resistance of the coatings. During the metallurgical preparation process of the coatings for the SEM observation, the procedure of the vacuum impregnation of epoxy for fastening the coating was deliberately obviated in the present investigations. In this circumstance, the unmelted particles imbedded in the coatings would be easy to be detached during the process of grinding and polishing due to their lower mechanical resistance, leaving behind the cavities and the remaining grains inside the individual unmelted particles that were not detached. The agglomerations of the inner grains inside the unmelted particles could not only be observed by inspecting inside the cracks, but also could be distinguished easily on the surface of the cross-sectional plane of the coatings by the obvious contrast between the granular shape of these grains agglomerations with the smooth and compact surroundings. From the area percentages of the cavities and porosities on the SEM images of the coating microstructure, which have been measured and listed in **Table 6-2**, the proportion of the unmelted particles and some coating properties such as the density, hardness, and adhesion/cohesion strength could be estimated.

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Table 6-1 Spraying conditions for coating microstructure investigations.

Cases			Plasma Gas		Arc Current <i>Ampere</i>	Carrier Gas		Powder Feed Rate		Figure Number
			<i>psi</i>	<i>slm</i>		<i>psi</i>	<i>slm</i>	<i>rpm</i>	<i>g/min</i>	
One Port	Influence of Plasma Gas	I 1	40	41.47	850	40	5.22	2.2	20	6.4
		I 2	60	56.85	850	40	5.22	2.2	20	6.5
		I 3	80	72.23	850	40	5.22	2.2	20	6.6
	Influence of Carrier Gas	I 7	60	56.85	850	20	3.16	2.2	20	6.7
		I 2	60	56.85	850	40	5.22	2.2	20	6.5
		I 8	60	56.85	850	60	7.28	2.2	20	6.8
Two-Port	Influence of Plasma Gas	II 1	40	41.47	850	30	4.19	2.2	20	6.9
		II 2	60	56.85	850	30	4.19	2.2	20	6.10
		II 3	80	72.23	850	30	4.19	2.2	20	6.11
	Influence of Carrier Gas	II 7	60	56.85	850	10	2.13	2.2	20	6.12
		II 2	60	56.85	850	30	4.19	2.2	20	6.10
		II 8	60	56.85	850	50	6.25	2.2	20	6.13

Table 6-2 Area percentages of the cavities and porosities of the coating microstructures investigated.

Cases			Figure Number	Area Percentage of Cavities and Porosities
One Port	Influence of Plasma Gas	I 1	6.4	18.69%
		I 2	6.5	21.94%
		I 3	6.6	29.39%
	Influence of Carrier Gas	I 7	6.7	16.84%
		I 2	6.5	21.94%
		I 8	6.8	24.17%
Two-Port	Influence of Plasma Gas	II 1	6.9	22.88%
		II 2	6.10	25.93%
		II 3	6.11	27.67%
	Influence of Carrier Gas	II 7	6.12	28.08%
		II 2	6.10	25.93%
		II 8	6.13	15.18%

6.3.1 Influence of Increasing Plasma Gas Pressure on Coating Microstructure for One-Port Internal Injection of Powders

For the coatings sprayed with one-port powder injection scheme, increasing the plasma gas flow rate from 41.47 *slm* to 72.23 *slm* lead to an increase in the area percentage of the cavities and the grain agglomerations from 18.69% to 29.39%, which could be observed by comparing **Figures 6.4 to 6.6**. As demonstrated earlier by the PDA measurements of the in-flight particle parameters, increasing the plasma gas flow rate brought the pronounced increase to the particle velocities. Particles with the increased velocities would penetrate through the core region of the plasma jet more rapidly with a shorter residence time. In this circumstance, more particles would be expected to experience insufficient heating and remain unmelted or partially melted upon impacting on the substrate. On the other hand, the increase of the plasma gas flow rate resulted in an overall decrease of the plasma jet temperature, which further reduced the heat transfer from the plasma jet to the in-flight particles and thus caused insufficient heating to more particles. Therefore, with the increased number of partially melted particles being imbedded inside the coating, the coatings produced under the higher plasma gas flow rate would be expected to exhibit a reduced density and hardness, with lower adhesion/cohesion strength and lower mechanical resistance.

6.3.2 Influence of Increasing Carrier Gas Pressure on Coating Microstructure for One-Port Internal Injection of Powders

The influence of the carrier gas flow rate on the coating microstructure could be observed by comparing **Figures 6.5, 6.7 and 6.8**. The coating produced under the 3.16 *slm* carrier gas flow rate obtained the lower area percentage of the cavities and the grain agglomerations of 16.84% as shown in **Table 6-2**, indicating that more particles adhering on the substrate were fully melted in this circumstance. According to the PDA

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measurements, both the mean particle velocities and diameters under the 3.16 *slm* carrier gas flow rate condition were lower than those under the higher carrier gas flow rate conditions of 5.22 *slm* and 7.28 *slm*, which favored the particle heating inside the plasma jet for the reason that particles with lower velocities had longer residence time inside the core region of the plasma jet at the same time, smaller particles were much easier to be heated due to their lower heat capacities. While the coatings produced under the lower carrier gas flow rate exhibited relatively tight connection with the underlying substrate, it could be observed that a continuous crack existed along the interface between the coating and substrate for the coatings sprayed with 7.28 *slm* carrier gas flow rate. This phenomenon could be explained by the reason that the first layer deposited on the substrate consisted too many partially melted particles under the too high carrier gas flow rate condition, which resulted in very poor connection between the coating and the underlying substrate. The bombardment of the following particles on the first layer made it even looser. In addition, the poor adhesion of the coatings with the substrate was further deteriorated by the mismatch in the thermal expansion of the top ceramic coating and the underlying metal substrate. Thus, the interface layers of the coating with the substrate had much lower mechanical resistance and would be much easier to be detached.

6.3.3 Influence of Increasing Plasma Gas Pressure on Coating Microstructure for Two-Port Internal Injection of Powders

The variations of the coating microstructures with respect to the plasma gas flow rate under the two-port powder injection scheme followed the similar trend as those under the one-port injection scheme, as demonstrated by comparing **Figures 6.9 to 6.11**. Increasing the plasma gas flow rate from 41.47 *slm* to 72.23 *slm* resulted in the increase of the area percentage of the cavities and the grain agglomerations on the coating microstructure images from 22.99% to 27.67%. This is because the higher velocities and larger particles

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penetrating inside the plasma jet under the higher plasma gas flow rate condition were disadvantageous for the particle heating. In this circumstance, the number of partially melted particles imbedded inside the coating would be increased.

6.3.4 Influence of Increasing Carrier Gas Pressure on Coating Microstructure for Two-Port Internal Injection of Powders

Comparing between **Figures 6.10, 6.12 and 6.13** reflected the influence of the carrier gas flow rate on the coating microstructures under the two-port powder injection conditions. It was obvious that the coating sprayed under the lower carrier gas flow rate of 2.13 *slm* exhibited extremely poor microstructure with too high percentage of unmelted particles or grains imbedded inside. The cracks appearing along the interface between the top coating and the underlying substrate was so extensive that it could be expected that the adherence of the coatings with the substrate was very poor and the coating would be easily peeled off from the substrate. Increasing the carrier gas flow rate to 6.25 *slm* resulted in a coating of much denser with lower area percentage of the cavity and the grain agglomerations as well as good connection of the top coating with the underlying substrate. The variation trend of the coating microstructures with respect to the carrier gas flow rate under the two-port powder injection conditions appeared to be contrary to that for one-port injection scheme. However, it should be reminded that for the two-port injection of powder, the carrier gas flow rate applied to each of the two injectors were approximately only half that indicated on the parameter control board of the plasma spray system. As mentioned earlier in the PDA measurements, the lower carrier gas flow rate in the two-port powder injection conditions would cause only the larger particles to be penetrate inside the plasma jet. These larger particles would most probably be only surface-melted upon impacting on the substrate, resulting in a coating that consisted of more unmelted particle grains and exhibited much lower mechanical resistance. It could be observed that the coating

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produced under the higher carrier gas flow rate of 6.25 *slm* for the two-port powder injection scheme was similar with the coating produced under 3.16 *slm* carrier gas flow rate condition for the one-port powder injection scheme. Thus, by comparing the influence of the carrier gas flow rate on the coatings sprayed under one-port and two-port scheme, it could be deduced that for the one-port injection of powders, the carrier gas flow rate lower than 3.16 *slm*, which was not investigated in the present experiments, would produced a coating with less density and more area percentage of the cavities and the unmelted grain agglomerations.

6.4 Measurement of Coating Deposition Efficiency

As mentioned above, a coating was formed by the successive building-up of the melted or partially melted particles on the substrate or pre-deposited layers. Depending on its in-flight characteristics prior to impacting, a particle would either stick on the substrate to form an adhered splat or bounce away from the substrate without contribution to the deposit. The quantity termed deposition efficiency (DE) indicated the percentage of particles that formed splats within an ensemble of particles arriving at the substrate. Deposition efficiency was one of the most important parameters with respect to the cost of producing a plasma-sprayed coating, especially when spraying the expensive materials. Thus, for the industrial applications of the plasma spraying, the spraying parameters would always be optimized to the deposition efficiency of the coatings provided that the desired coating properties could be satisfied.

In this section, the variations of the coating deposition efficiency and the print offset of the deposits with the spraying parameters (**Table 6.3**) were systematically investigated. In addition, efforts were devoted for the first time to studying and trying to establish a relationship between the coating deposition efficiency and the PDA measured particle concentration and volume flux. The reason for relating the coating deposition efficiency to the PDA measured particle concentration and volume flux was based on the fact that both of them were sensitive to and would change with the amount of melted or surface-melted particles at impact.

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Table 6-3 Parameters for the investigation of their influence on the coating deposition efficiency and the position of deposit strips.

<i>Case</i>	<i>Number of Port</i>	<i>Parameter of Interest</i>	<i>Parameter Range</i>	<i>Figure</i>
1	1	Plasma Gas Flow Rate (<i>slm</i>)	41.47-80	6.15 (a)
2	1	Arc Current Level (<i>Ampere</i>)	400-900	6.15 (b)
3	1	Carrier Gas Flow Rate (<i>slm</i>)	3.16-8.5	6.15 (c)
4	2	Plasma Gas Flow Rate (<i>slm</i>)	41.47-80	6.16 (a)
5	2	Arc Current Level (<i>Ampere</i>)	400-900	6.16 (b)
6	2	Carrier Gas Flow Rate (<i>slm</i>)	3.16-8.5	6.16 (c)

The deposit stripes for deposition efficiency measurements were sprayed onto the same substrate pieces for producing the coating mentioned previously. Each substrate was placed at the standoff distance of 80mm from the nozzle exit. Manipulated by the computer-controlled 3-D traversing rig mentioned earlier, the centerline of the plasma torch was adjusted to aim at the midline between the two holes, and then was traversed horizontally, without vertical movement of the plasma torch, with a speed of 10cm/s through the substrate for 10 passes.

Subsequently, each deposit stripe was brought to the CMM machine for surface profile scanning along its vertical direction to accurately locate the position of the thickest point and its relative distance from the midline of the two holes on the substrate. Due to the gradual transition of the coverage percentage of the deposit on the substrate, as shown by a macroscopic view of a representative deposit stripe taken by a digital camera in **Figure 6.14**, it was impossible to give an unambiguous determination of the top and bottom borderlines of the deposit stripe. Assuming the thickness of the deposit stripe roughly follows a Gaussian distribution, the two borderlines were here defined as the positions where the thickness of the deposit stripe was $1/e^2$ of the maximum thickness. Thus, the relative position of the deposit stripes with respect to the aim point of the plasma torch, together with the width of them could be determined for each spraying condition.

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The substrates were weighted before and after deposition to obtain the deposit weight gains by a balance whose accuracy was 10^{-6} g. The deposition efficiency represents the mass ratio of deposited particles to total sprayed particles, which was determined by

$$DE = G_d V_s / L_s F N \quad (6-1)$$

where G_d is the deposit weight gain, V_s the traverse speed of the substrate, L_d the length of the deposit stripe, F the powder feed rate and N the number of passes.

6.4.1 Influence of Increasing Plasma Gas Pressure on Coating Deposition Efficiency for One-Port Internal Injection of Powders

Figure 6.15 (a) illustrated the variation of the deposition efficiency and the position of the deposit stripe with the plasma gas flow rate for the one-port powder injection condition. The height of the bars represented the width of the individual deposit stripes and their positions relative to the left y-axis represented the relative position of the individual deposit stripes respect to the aim point of the plasma torch. It was shown that increasing the plasma gas flow rate lead to a decrease of the deposition efficiency, which indicated a decreased number of particles being adhered on the substrate with a higher plasma gas flow rate. This finding was in good agreement with that observed in the coating microstructure analysis, where the increase of plasma gas flow rate resulted in a higher area percentage of the cavities and the unmelted grain agglomerations. The reason was attributed to the increased number of unmelted particles in the higher plasma gas flow rate conditions. The position of the deposit stripe was found to shift towards the upper portion of the substrate, i.e. the top side of the plasma torch, and the width of the deposit stripe was shortened with increasing the plasma gas flow rate. This phenomenon could be caused by the density and velocity increase of the plasma jet due to the increase of the plasma gas flow rate. In this case, the particle penetration distance inside the plasma jet would be shortened as mentioned in the previous section for the PDA measurements of the in-flight

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mean particle velocity and diameter distributions. Thus, they would impact on the substrate at the upper position with a lower particle dispersion, i.e. shortened width of the deposit stripe.

It was of interest that the variation trend of the deposit patterns with the plasma gas flow rate could be reflected by the PDA measured radial distributions of the particle concentration and volume flux, as previously illustrated in **Figure 5.20** in **Chapter 5**. Increasing the plasma gas flow rate lead to the decrease of the particle concentration and volume flux, and their distribution curves shifted towards the left side of the plasma jet, which corresponded to the top side of the plasma torch in producing the deposit stripes. As mentioned in **Chapter 5**, the particles that satisfied all the validation criteria for the PDA measurements and were included in the PDA concentration and volume flux calculations were most probably in the molten or surface-melted state. In another word, these particles validated by the PDA measurement were just those that contributed most to the actual deposit formation. Thus, when altering the spraying parameters, the variations in the number of in-flight molten or semi-molten particles upon impact were not only reflected by the deposition efficiency of the deposit, but also by the PDA measured particle concentration and volume flux in the same tendency. The position of the top and bottom borders of the deposit stripes relative to the plasma torch aim point corresponded to the left and right edges of the particle stream relative to the centerline of the plasma jet where the data rate of the PDA measured particles approached zero. It should be noted that due to the difficulty in finding the accurate top and bottom borderlines of the deposit stripe, they were defined as the position where the thickness of the deposit stripe was $1/e^2$ of the maximum thickness. Thus, the width of the deposit stripe measured was somewhat shorter than the distance between the corresponding left and right edges of the particle stream measured by

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the PDA. However, the position of the deposit stripe was in consistence with that of the particle stream reflected in the PDA measured radial distribution of the particle concentration and volume flux.

6.4.2 Influence of Increasing Arc Current Level on Coating Deposition Efficiency for One-Port Internal Injection of Powders

The similar trend between the variations of deposit patterns and the PDA measured radial distribution of particle concentration and volume flux with the arc current level could also be found in **Figure 6.15 (b)** and **Figures 5.21**. Increasing the arc current level brought significant increase to the deposition efficiency without notable alteration to the width and position of the deposit stripe, and the PDA measured radial distributions of the particle concentration and volume flux also reflected the same trend. Although it has been demonstrated earlier by the PDA measurement that increasing the arc current level lead to an increase to the in-flight particle velocity, the overall particle temperature increase at the same time must have counteracted the effect of the shorter residence time of the particles inside the plasma jet with higher velocities and caused increased number of particle being melted.

6.4.3 Influence of Increasing Carrier Gas Pressure on Coating Deposition Efficiency for One-Port Internal Injection of Powders

The influence of the carrier gas flow rate on the deposit patterns was illustrated in **Figure 6.15 (c)**. It could be observed that increasing the carrier gas flow rate from 3.16 *slm* onwards lead to a decrease of the deposition efficiency, which was also consistent with the coating microstructure analysis. The deposit stripe shifted towards the powder injection direction, i.e. the lower position of the substrate, with increasing the carrier gas flow rate due to the increased penetration distance of the particles with higher injection velocities.

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Referring to the PDA measurements under the same spraying conditions in **Figure 5.22** in **Chapter 5**, it could be shown that the particle concentration and volume flux also decreased with increasing the carrier gas flow rate and the radial distribution curves shifted towards the powder injection direction in the similar way exhibited by the coating deposition efficiency and spraying patterns.

6.4.4 The Respective Influence of Increasing Plasma Gas Pressure, Arc Current Level and Carrier Gas Pressure on Coating Deposition Efficiency for Two-Port Internal Injection of Powders

Figure 6.16 illustrated the respective influence of the plasma gas flow rate, carrier gas flow rate and arc current level on the deposit patterns for the two-port internal powder injection conditions. Similar with the corresponding PDA measured radial distributions of particle concentration and volume flux shown in **Chapter 5**, the deposit stripes were almost symmetrically distributed about the centerline of the plasma torch. Increasing the plasma gas flow rate resulted in the decrease in not only the deposition efficiency (**Figure 6.16 (a)**) but also the overall particle concentration and volume flux measured by the PDA, which was in consistence with the coating microstructure analysis. At the lower plasma gas flow rate, two peak regions of particle concentration and volume flux occurred in the distribution curves, thanks to the lower plasma jet momentum that allowed most of the particles injected through each of the two injectors to penetrate through the plasma jet and concentrate on the opposite side of their respective injector. Increasing the plasma gas flow rate thwarted the particle penetration inside the plasma jet, which made the number of particles concentrated around the centerline higher than that at other locations, at the same time lead to a shortened dispersion of the particles and thus a decrease in the width of the deposit stripe. Increasing the arc current level resulted in the pronounced increase to both the deposition efficiency (**Figure 6.16 (b)**) and the overall PDA measured particle

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concentration and volume flux. The width of the deposit stripe increased somewhat with the arc current, which might be due to the increased number of the melted particles in the broader range of the plasma jet. Finally, increasing the carrier gas flow rate enlarged the dispersion of the particles and the width of the deposit stripe increased gradually, as shown in **Figure 6.16 (c)**. The variation of the deposition efficiency with the carrier gas flow rate was also a reflection of the coating microstructure. As demonstrated in **Section 6.3**, the carrier gas flow rate set as 3.16 *slm* produced a coating of very poor microstructure with high area percentage of the cavities and the unmelted grain agglomerations. Correspondingly, in this condition the deposition efficiency was also found to be very low. Increasing the carrier gas flow rate increased the deposition efficiency gradually, until reached the maximum value of 33% at the 6.25 *slm* carrier gas flow rate. After that, the deposition efficiency dropped with further increasing the carrier gas flow rate, but the decrease was not as steep as that for the one-port injection condition. These findings were in good agreement with the coating microstructure analysis, and were also reflected by the radial distributions of the particle concentration and volume flux measured by the PDA.

6.5 Summary

Corresponding to the individual spraying conditions for the PDA measurement in the previous chapter, the microstructure and deposition efficiency of the sprayed coatings were characterized to make a comparison with the PDA measured in-flight particle velocity, diameter, concentration and volume flux distributions. Through the comparison of the in-flight particle characteristics and the off-line deposit analysis, conclusions could be drawn that it was the particle temperature at impact that had the most predominant influence on the coating microstructure and the deposition efficiency. The increase in the spraying parameters that brought solely to the particle velocity increase such as in the case of increasing the plasma gas flow rate would lead to increased number of unmelted particles, which would be reflected on the deterioration of the coating microstructure and the decrease of the deposition efficiency. Increasing the arc current level increased the particle velocities, but it was obvious that the overall particle temperature increase at the same time would overwhelmingly counteract the effect of the particle shorter residence time inside the plasma jet resulted by the increased particle velocity, thus led to increased number of particle being melted. The carrier gas flow rate determined the initial particle injection momentum with respect to that of the plasma jet, and it must be chosen carefully to ensure the maximum number of particles experience the longest residence time in the hottest zone of the plasma jet. Too low or too high of the carrier gas flow rate would result in a coating with poor quality and decreased deposition efficiency. Finally, it was confirmed by the PDA measurements that the powder feed rate had no remarkable influence on the particle velocity and diameter distributions within the investigated range.

Particles injected through the opposing injectors would result in more symmetrical distribution inside the plasma jet, which was believed to be beneficial for producing a

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coating with more uniform microstructure and thickness. The deposition efficiency of the coating would also be slightly increased with the two-port powder injection scheme.

The most interesting finding was that the PDA measured radial distribution of the particle concentration and volume flux could closely reflect the variation of the coating microstructure and deposition efficiency. Although it was unanimously agreed that the particle velocity and temperature at impact were the most important parameters determining the coating properties, their influences on the coating properties were quite complicated and could only be qualitatively estimated. Thus, the variation of the coating properties could hardly be predicted from the particle velocity and temperature information directly and accurately. Thanks to its unique measurement requirement of the PDA, the particles validated for the concentration and volume flux measurements were most probably in the molten or surface-melted state, and it were just these particles that contributed most to the actual deposit formation. Therefore, the variation of the PDA measured particle concentration and volume flux was consistent with that of the coating properties.

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Figure 6.1 The segments of coatings imbedded inside a molding for the microstructure analysis.

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Figure 6.2 The SEM images of (a) a splat formed by the fully melted YSZ particle and (b) a fully melted YSZ particle but solidified before impacting on the substrate.

Figure 6.3 The SEM images of (a) an unmelted YSZ particle and (b) a partially surface melted YSZ particle showing the internal agglomeration grains.

Figure 6.4 The SEM image of the coating microstructure produced under 41.47slm plasma gas flow rate, one-port internal injection of powder (case I1 in **Table 5-2**).

Figure 6.5 The SEM image of the coating microstructure produced under 56.85slm plasma gas flow rate, one-port internal injection of powder (case I2 in **Table 5-2**).

Figure 6.6 The SEM image of the coating microstructure produced under 72.23slm plasma gas flow rate, one-port internal injection of powder (case I3 in **Table 5-2**).

Figure 6.7 The SEM image of the coating microstructure produced under 3.16slm carrier gas flow rate, one-port internal injection of powder (case I7 in **Table 5-2**).

Figure 6.8 The SEM image of the coating microstructure produced under 7.28slm carrier gas flow rate, one-port internal injection of powder (case I8 in **Table 5-2**).

Figure 6.9 The SEM image of the coating microstructure produced under 41.47slm plasma gas flow rate, two-port internal injection of powder (case III in **Table 5-3**).

Figure 6.10 The SEM image of the coating microstructure produced under 56.85slm plasma gas flow rate, two-port internal injection of powder (case II2 in **Table 5-3**).

Figure 6.11 The SEM image of the coating microstructure produced under 72.23slm plasma gas flow rate, two-port internal injection of powder (case II3 in **Table 5-3**).

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Figure 6.12 The SEM image of the coating microstructure produced under 2.13slm carrier gas flow rate, two-port internal injection of powder (case II7 in **Table 5-3**).

Figure 6.13 The SEM image of the coating microstructure produced under 6.25slm carrier gas flow rate, two-port internal injection of powder (case II8 in **Table 5-3**).

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Figure 6.14 The example photo of the deposit stripe.

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Figure 6.15 The position of the deposition stripes relative to the torch horizontal centerline and the deposition efficiencies under different (a) plasma gas flow rate, (b) arc current and (c) carrier gas flow rate for one-port internal injection of powder.

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Figure 6.16 The position of the deposition stripes relative to the torch horizontal centerline and the deposition efficiencies under different (a) plasma gas flow rate, b) arc current and (c) carrier gas flow rate for two-port internal injection of powder.

Chapter 7

Conclusions and Future Works

7.1 Conclusions

Investigations on the plasma spraying process relied mostly on the non-intrusive optical diagnostic tools due to the hostile high temperature and speed environment. Among the various diagnostic methods, the Phase Doppler Anemometry (PDA) has been widely recognized as one of the most sophisticated and well-established technique for simultaneous measurement of the in-flight particle velocities and diameters. However, optimization of the PDA configurations and careful interpretation of the measurements according to the specific applications is critical for the effectiveness of the PDA technique. Although the PDA technique has ever been applied in measuring the plasma sprayed particle velocities and diameters, a detailed assessment on its reliability and accuracy under the plasma spraying condition has seldom been mentioned before.

The present works have been focused on investigating the particular response of the PDA to the plasma sprayed particles, and the correlations between the PDA measured particle in-flight characteristics and the sprayed coating/deposit properties. When using the PDA technique to measure the plasma sprayed particles, error sources such as the Gaussian effect and Slit effect that would be encountered in measuring many poly-phase flows could be totally avoided since the particles for plasma spraying are non-transparent and only the reflected light from the particles could be detected by the PDA receivers. The receiving angle of the PDA receivers, which is one of the most important parameters influencing the

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PDA measurement, could be compromised with the experimental set-up as long as the PDA measurements can be maintained an acceptable signal-to-noise ratio. The possibility of the multi-particle coincidence inside the PDA measurement volume has been numerically estimated by the Poisson statistics equations and was found to be very low, which indicated that the phenomenon of two or more particles appearing simultaneously in the PDA measurement volume was rare under the plasma spray condition. It only played a minor role on the PDA rejection of particle. The PDA measurement was very sensitive to the particle surface condition, which was proved by the waveforms of the Doppler bursts originated from the water droplet (perfect spherical shape and smooth surface), the plasma sprayed NiCrAlY particle (in molten state with spherical shape and smooth surface), and the plasma sprayed YSZ particle (unmelted with non-spherical shape and rough surface). The waveforms for the former two particles exhibited a form of regular Doppler burst, while that for the last one was characterized by random indented distribution. Through measuring the NiCrAlY particle samples with known size ranges subjected to different spraying conditions to achieve different level of surface roughness and sphericity, it was further demonstrated that the rejection of the particles and the miscalculation of the particle diameter by the PDA were predominantly caused by the rough surface and irregular shape of the particles. For the cases of measuring the samples with increased number of unmelted particles, the particle diameter distribution became broader even though the particle mean diameter still fell within the size range of the individual samples. This phenomenon indicated that some particles validated for the diameter measurement by the PDA were actually recorded with a wrong diameter, but they were only the minority of all the diameters measured. Although the miscalculated particles had little influence on the particle mean diameter, the smaller particles calculated as larger ones by mistake would have significant effect on the PDA measured volume flux. Through the comparison of the

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PDA measured YSZ particle volume flux with the volume of the corresponding actual deposit, it was demonstrated that the particle volume flux distribution obtained directly from the PDA measurement was greatly affected by the existence of these miscalculated particles. However, the modified volume flux distribution obtained by filtering these larger miscalculated particles exhibited a surface profile and total volume value closer to those of the actual deposit. The variation of the local thickness distribution and the total volume of the deposit with the plasma spraying parameters might also be approximately estimated by the PDA measured modified particle volume flux distribution.

With the proven ability to accurately measure the diameter and volume flux of the plasma sprayed particle with spherical shape and smooth surface, i.e. the particles in molten state, the PDA was consequently used to characterize the variation of the radial distribution of the particle mean velocity, diameter, concentration and volume flux under different plasma spraying conditions, which included the different levels of plasma gas flow rate, arc current level, carrier gas flow rate, and powder feed rate for the powder injected through different arrangements of the powder injector. All the PDA obtained particle information underwent post-processing by filtering the particles whose diameters measured by the PDA obviously exceed the actual maximum particle diameter. The particle in-flight characteristics were then compared with the microstructure of the coatings and the deposit pattern under the same spraying conditions. It was inferred that for spraying materials with high melting point such as the YSZ powders, the particle temperature upon impacting on the substrate had the most important influence on the coating microstructure and the deposition efficiency. Increasing the plasma gas flow rate increased the particle velocity significantly. However, with the shortened residence time of the particles inside the plasma jet and the lower heat transfer from the plasma to the particles, the particle temperature

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would be expected to be reduced, which could be reflected by the poor coating microstructure and decreased deposition efficiency under higher plasma gas flow rate condition within the range investigated. Increasing the arc current level increased the particle velocity and temperature simultaneously but led to the improved coating quality and increased deposition efficiency. The appropriate selection of the carrier gas flow rate was of utmost importance since too low or too high of the carrier gas flow rate would result in a coating with poor quality and decreased deposition efficiency. Finally, it was confirmed by the PDA measurements that the powder feed rate had no remarkable influence on the particle velocity and diameter distributions within the investigated range. In addition, it was found that particles injected through the opposite two injectors would be more symmetrically distributed inside the plasma jet, which was believed to be beneficial for producing a coating with more uniform microstructure and thickness. The deposition efficiency of the coating would also be slightly increased with the two-port powder injection scheme.

Comparing the variation of the PDA measured radial distribution of the particle concentration and volume flux with that of the coating microstructure and deposition efficiency indicated that there existed high consistency between them due to the reason that they were all very sensitive to the amount of particles being melted. The particles validated for the concentration and volume flux measurements by the PDA were most probably in the molten or surface-melted state, and it were just these particles that contributed most to the actual deposit formation. Therefore, the variation of the PDA measured particle concentration and volume flux was in consistence with that of the coating properties.

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In summary, this thesis demonstrated the reliability of the PDA technique in characterizing the in-flight plasma sprayed particle behaviors and revealed for the first time that there existed a distinct and consistent relationship between the PDA measured particle concentration or volume flux with the coating microstructure and deposition efficiency. Restricted by the resolution and accuracy of the current particle temperature measurement devices, the variation of the coating properties usually could not be predicted reliably from the particle temperature information. However, the PDA measured particle concentration or volume flux would provide an alternative to quantitatively assess the variation of the coating microstructure and the deposition efficiency. By monitoring the in-flight particle concentration or volume flux variations, instead of the particle velocity and temperature, the optimal settings of the plasma spraying parameters could also be determined conveniently and reliably.

7.2 Recommendations for Future Works

The PDA technique has proven its superior capability in effectively measuring the plasma sprayed particles in this thesis. However, it has also been demonstrated that the particle information obtained directly from the PDA software must undergo post-processing to acquire the reliable measurement result. In the present investigations, the post-processing method was relatively simple by filtering the particles whose diameter exceeded the maximum expected-to-be-correct particle diameter by the PDA. A more sophisticated and rigorous method should be developed to accurately interpret the PDA measured particle information in the future, which is expected to be able to further reduce the measurement error and more reliably represent the in-flight particle behaviors.

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In this thesis, the PDA measured in-flight particle characteristics have been compared with the coating microstructure and deposition efficiency. In the future, other coating properties such as the porosity, adhesion/cohesion strength should also be investigated.

In addition, the conclusions about the influence of the plasma spraying parameters on the in-flight particle characteristics as well as the coating microstructure and deposition efficiency were drawn for the high melting point YSZ powders. For the low melting-point metal powders, similar investigation should be performed to examine the validity of these conclusions.

Finally, the comparisons of the experimental results and simulation work are also recommended to carry out to improve the numerical modeling of the plasma spraying process.

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Appendix I

Extended PDA Systems

Several modified optical configurations to the conventional PDA have been developed to overcome the Gaussian beam defect and Slit effect. Two more recent suggestions appear to be more practical from an instrumentation point of view. The first of these is the planar PDA (PPDA), named for the fact that the detectors lie in the plane of the intersecting laser beam. Although both reflective and refractive scattered light is received by the PPDA, the contributions come with a time-delay and furthermore the refractive contribution is much higher in amplitude. Thus a clear distinction at the signal detection stage can be made to eliminate sizing errors. By processing the half part of signal burst with constant phase difference, the system can be freed from the Gaussian beam defect. The second method of eliminating trajectory effect when using a two-component system, is to change the flow direction of droplets perpendicular to the beam intersection plane, that is, parallel to the fringes, termed a modified standard PDA (MS PDA). The measurement principle of the MS PDA is almost the same as PPDA, with the exception that another pair of laser beams whose bisecting plane is perpendicular to the particle movement direction is needed to measure the particle main flow velocity. However, both the PPDA and MS PDA suffer from the constraint that the flow must be aligned either with the x or y axis respectively. In a turbulent flow, trajectory errors can still be expected with these systems. A promising method which can minimize or eliminate the trajectory errors disregarding the main flow movement direction was proposed with the name Dual Mode PDA. It combines two detectors of a standard system with two detectors of a planar system in a single receiving unit with the aim of simplifying alignment. By

comparing the measured size using a standard PDA system with the simultaneous size measurement of a planar PDA system, it can provide reliable particle size information.

AI.1 Planar PDA

Numerical studies have shown that the Gaussian beam defect is not symmetrical about the y -axis. **Figure AI.1** presents a numerical simulation result based on the generalized Lorenz-Mie theory for different locations of a $40\ \mu\text{m}$ water droplet in an $80\ \mu\text{m}$ probe volume, presented as a phase difference map. The computed phase difference is constant for $y > 0$, but is strongly dependent on y for $y < 0$. **Figure AI.1** also indicates that there is almost no phase difference dependence on the x location. The iso-phase difference lines are parallel to the x -axis and, hence, perpendicular to the LDA fringe.

Figure AI.1 Simulated phase difference map, $d_p = 40\ \mu\text{m}$; $\theta/2 = 0.9^\circ$; $\alpha = 30^\circ$; $\psi_{1,2} = \pm 4.1^\circ$.

Based on these studies, a new PDA optical set-up was proposed recently aimed at a complete elimination of the Gaussian beam defect. **Figure AI.2** shows the proposed optical set-up which can be referred as a planar layout: the two laser beams, their

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polarization directions and the two detectors are all in the xz plane. The elevation angles ψ_1 and ψ_2 of the detectors have about the same magnitude as the off-axis angle in a conventional layout. A numerical investigation on this optical set-up for a water droplet of $52\ \mu\text{m}$ is presented in **Figure AI.3 (b)** as a map of the phase difference between the two elementary detectors in the detection unit. It can be seen that the iso-phase difference lines are rotated by 90° compared with **Figure AI.1** and, hence, are parallel to the LDA fringes. In this way the signal phase is no longer a function of location y but becomes a function of location x for $x > 0$. Physically, this means that although the Gaussian beam effect cannot be removed, its influencing zone is moved from the half probe volume with $y < 0$ to the locations with $x > 0$. If a particle crosses the LDA fringes along a trajectory parallel to the x axis from negative to positive x locations, the front part of the corresponding Doppler burst is coming from the half probe volume with $x < 0$ where the intensity gradient of the incident Gaussian beams has no influence on the signal phase. By processing this part of the signal only, the system can be freed from the Gaussian beam defect.

Figure AI.2 Sketch of Planar PDA

Figure AI.4 Optical geometry of a modified standard PDA

The concept of modified standard PDA was first introduced by Xu and Tropea (1994), in which they provided theoretical analysis and experimental verification. They applied the Generalized Lorenz-Mie theory (GLMT) program of Grehan et al. (1992) to a $38\text{ }\mu\text{m}$ water droplet in a measurement volume with diameter of $50\text{ }\mu\text{m}$. The scattering characteristics as a function of position in the measurement volume are given by the phase difference and intensity maps, as shown in **Figure AI.5**. These are representative for other droplet sizes and indicate that for trajectories parallel to the y axis the phase difference measured at the detectors will remain constant for positive values of y , but vary strongly in regions of negative y , i.e. the side of the measurement volume opposite the detectors. Furthermore, the phase difference obtained for positive y values agrees well with the phase difference predicted by geometric optics for refractive dominated scattering.

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For particles moving parallel to the y axis, the intensity of the scattered light attains a maximum at approximately $18\ \mu\text{m}$ (about 47% of the droplet diameter) offset from the middle of the volume in the positive y direction. The location at which the signal amplitude reaches a maximum therefore also corresponds to a region in which the phase difference is expected to be constant. A processor that detects the highest signal amplitude and performs the phase estimation on this portion of the signal will therefore yield a reliable size measurement.

For the modified standard PDA system with refracting mode, the time delay between different contributions reaching the detectors was used to suppress the trajectory effect by processing only the portion of the burst originating from refractive scattering. However, later this method was further developed to make use of all portion of the signal to extract the refractive indices of particles. The principle is that, of the two scattering contribution, i.e. reflection ($p=0$) and refraction ($p=1$), only the refracted light is influenced by the material properties of the particle. The phase of the refracted light from a particle is a function of the optical set-up, particle size and particle refractive index. The phase of the reflected light is, however, only a function of optical set-up and particle size and does not depend on the particle refractive index. From the reflected burst, the particle diameter can be deduced. Knowing this diameter and the optical parameters of the phase-Doppler set-up, the particle refractive index can be determined from the phase of the refracted burst.

Figure AI.5 Contour maps of equal phase difference ($^{\circ}$) and intensity ($\log(mW)$) for a $38\text{ }\mu\text{m}$ water droplet in a $50\text{ }\mu\text{m}$ measurement volume $\lambda = 514.5\text{nm}$, $\theta/2 = 2.309^{\circ}$, $\alpha = 70^{\circ}$, $\psi_{1,2} = \pm 4.096^{\circ}$. The indicated trajectory would lead to an error of 13% in the size measurement.

Clearly, the biggest restriction for the modified standard PDA is that this configuration requires a two-component PDA system. For a one-component set-up, only the cross-stream velocity component of the particles would be acquired, which, in general, is of less interest. Additional pair of laser beams with the bisecting plane perpendicular to the main flow direction is needed.

AI.3 Dual Mode PDA

As mentioned before, either the planar PDA or the modified standard PDA has the inherent restriction that the flow must be aligned either with the x or y axis respectively. For measurements in complicated flows, the Dual Mode PDA has been developed.

The Dual Mode PDA is based on a four-beam, two-component system, which combines two detectors of a standard system (SPDA) with two detectors of a planar system (PPDA) in a single receiving unit. The optical layout is illustrated in **Figure AI.6**. The transmitting optics does not differ in any way from those of a conventional PDA system. Typically, the main flow direction is along the Y axis; however, deviations from this direction do not lead to errors, but at most a non-validation of the size measurement.

The receiving unit provides four apertures for the four detectors. Detectors V_1 and V_2 are for the PPDA and the velocity component aligned with the Y axis, whereas U_1 and U_2 are for the standard PDA and the velocity component aligned with the X axis. The detector pair U_1 - U_3 used in the conventional three-detector PDA to overcome the 2π ambiguity and to perform the sphericity check/inconsistency check is replaced with the planar PDA to recognize and reject bursts influenced by the Gaussian beam defect. Interference filters are used in front of the detectors to separate the scattered light from each of the beam pairs according to its color.

Figure AI.6 The optical layout of the Dual Mode PDA

AI.4 Three-Detector Fiber PDA

Sankar et al. (1992) have shown that the trajectory dependent sizing errors can be minimized by the proper selection of the optical parameters, namely, the beam intersection angle and using the two-detector-pair arrangement of PDA. This work represented the first recognition of the importance of the relative phase difference between the reflected and refracted light in resolving the trajectory dependent error problem. Increasing the beam intersection angle serves to increase the difference between the phase of light scattered by refraction and reflection, allowing a more effective discrimination using the phase ratio from the two pairs of detectors. The three detector Fiber PDA was also used in current investigations and the introduction of its measurement mechanism has been introduced before.

Appendix II

Principle of Particle Temperature Measurement

Particle temperature determination is based on two-color pyrometry. Optical temperature measurement is based on the well-known Planck's law of radiation, which states that objects emit electromagnetic radiation whose intensity and wavelength distribution depend on the temperature of the object. Even cold objects emit radiation, but when they are heated, the radiation is shifted to shorter wavelengths, i.e. to visible and near-infrared regions. When the temperature rises, object appears first dark red, changing then to brighter orange, finally reaching a bright white glow.

Planck's law of radiation gives the relationship between the temperature of an object and the total light flux (W) emitted by it.

$$W(T) = A\varepsilon(\lambda, Y)B(T) \quad (\text{AII-1})$$

where

$$B(T) = C_1 \int_{\lambda_0}^{\lambda_1} \frac{d\lambda}{\lambda^5 \left[\exp\left(\frac{C_2}{\lambda T}\right) - 1 \right]} \quad (\text{AII-2})$$

is the radiant exitance (the radiation emitted) for a black body (an idealised radiator) in the spectral range $[\lambda_0, \lambda_1]$. Here C_1 and C_2 are constants, λ is the wavelength and T is the temperature of the object.

A is the object's surface area and $\varepsilon(\lambda, Y)$ is called the emissivity. It describes the object's capability of emitting radiation compared to that of an idealised emitter (black

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body). $\varepsilon(\lambda, T) = 1$ is for a black body, but < 1 for all real world objects. In many cases the exact value of the emissivity is not known.

Figure AII.1 shows the light intensity expressed by **Equation AII-1** in relative units for an object made of tungsten in different temperatures. It can be seen that when the temperature rises, the total intensity rises rapidly. In addition, the radiation is shifted into shorter wavelengths.

The output signal of any physical light detector is proportional to W by a factor that depends on the light collecting optics and the detector sensitivity.

From **Equation AII-1** it can be seen that in addition to the temperature, the light flux emitted by an object on a certain wavelength depends both on the emissivity and size of the object. Thus when applied to the measurement of particles in thermal spraying, it is impossible to solve the particle temperature from **Equation AII-1** unless both the emissivity of the particle material and the particle size are known. This problem can be partially solved by the means of two-color pyrometry.

Two-color pyrometry is a method for optical temperature measurement that is based on the measurement of the light emitted by the object in two separate wavelength ranges with centre wavelengths λ_1 and λ_2 . The ratio of the radiation fluxes emitted in those wavelength ranges is now:

$$\frac{W_{\lambda_1}(T)}{W_{\lambda_2}(T)} = \frac{A\varepsilon(\lambda_1, T)B(T)}{A\varepsilon(\lambda_2, T)B(T)} = \frac{B_{\lambda_1}(T_c)}{B_{\lambda_2}(T_c)} \quad (\text{AII-3})$$

where T_c is the color temperature of the object. When the object is a gray body, i.e. its emissivity is independent of the wavelength, the color temperature is equal to the true

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temperature of the object. This is, however, the case for the most real world objects and materials.

A two-colour pyrometer is a device that measures the light emitted by the object in two wavelength ranges so that its color temperature T_c can be calculated from the calibration information:

$$\frac{R_1}{R_2} = \frac{F_1(T)}{F_2(T)} \rightarrow T \quad (\text{AII-4})$$

where R_1 and R_2 are the output signals of the pyrometer and F_1 and F_2 are system response functions determined by the calibration so that they depend on $B_{\lambda 1}$ and $B_{\lambda 2}$ in a known way. As seen also from **Equation AII-1**, the relative signal on measurement range 1 (the leftmost grayed area) increases compared to the measurement range 2 (the grayed area on the right) when the temperature rises.

Two-colour pyrometry eliminates the effects of particle size, emissivity (assuming the particle is a gray body, i.e. the emissivity does not depend on the wavelength) and non-ideal focusing of the optics on temperature measurement.

The most important systematic error source is the non-grayness, i.e. the wavelength dependency of the emissivity of the particle material. It can be considerable especially for metallic materials. If the ratio of the emissivities of the material in the measurement wavelength ranges is known, the error can be corrected. Unfortunately, the wavelength and temperature behavior of most thermal spraying materials are usually unknown.

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Figure AII.1 Relative intensity of an object made of tungsten in different temperatures.

Appendix III

List of Publications

1. P. Wang, S. C. M. Yu, and H. W. Ng, Particle Velocities, Sizes and Flux Distribution in Thermal Spray with Two Powder Injection Ports, 2nd International Conference on Spray Deposition and Melt Atomization and 5th International Conference on Spray Forming, Vol.2, (7) 57-68.
2. P. Wang, S. C. M. Yu, and H. W. Ng, Particle Velocities, Sizes and Flux Distribution in Plasma Spray with Two Powder Injection Ports, Materials Science and Engineering A, Vol. 383, Issue 1, (10) 2004, 122-136.