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Characterization of Cold Spray Titanium Supersonic Jet

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Titanium is widely used in aerospace, highly corrosive environments, and implants due to unique properties such as high strength to weight ratio and excellent corrosion resistance. Cold gas dynamic spray (cold spray) technology, in contrast to current fabrication technologies, has provided the potential for titanium to be utilized in broader industrial applications and at lower cost. Particle velocity is the most important parameter in the cold spray process that leads to successful deposition of titanium at supersonic speeds. In this study, particle image velocimetry (PIV) is utilized to characterize supersonic flow field for a commercially pure (CP) titanium powder. The results represent experimentally determined velocity for titanium particles under supersonic conditions with respect to propellant gas, spray temperature, and stagnation pressure. The high velocity flow region outside of the cold spray nozzle was significantly extended using helium. An increase in stagnation temperature results in a high velocity region close to the axis of the cold spray nozzle. In contrast, an increase in pressure expands the high velocity regions in the cold spray plume. The PIV that is a whole-flow-field process is a practical characterization technique for optimization of parameters and validation of the future models for the cold spray process.

Keywords cold spray, particle image velocimetry, supersonic jet, titanium

1. Introduction

Cold spray is a deposition process in which small particles in the solid state accelerate to high velocities (normally above 500 m/s) in a supersonic gas jet and deposit on the substrate material. The kinetic energy of the particles is used to obtain bonding through plastic deformation upon impact with the substrate. This provides a unique advantage for cold spray to be exploited for oxygen-sensitive materials such as titanium. The unique properties of titanium such as high strength to weight ratio, excellent corrosion resistance and bio-compatibility have made this material a favorable option for many applications in aerospace (Ref 1), implants (Ref 2), and corrosive environments (Ref 3). An earlier report by Karthikeyan (Ref 4) reveals that cold spray fabrication leads to reduction in material input, elimination of mold and melting cost, reduction of rework and finishing for titanium products, thus making this material an affordable choice for wider industrial applications.

Generally, successful supersonic deposition results in formation of a dense material (Ref 5, 6). To achieve optimal conditions for deposition, the information of supersonic jet within the nozzle, at the nozzle exit, and before deposition of particles is paramount which has been subject of many studies (Ref 7-10). Particle image velocimetry (PIV) is a promising technique that determines particle displacement over a short separation time. Gilmore et al. (Ref 11) in earlier studies have determined copper particle velocity in respect to a rectangular cold spray nozzle using a PIV technique. A recent study by Pattison et al. (Ref 12) investigates the effect of stand off distance and bow shock on aluminum, copper, and titanium. In this study PIV measurements of the cold spray free jet for helium at room temperature and nitrogen at 300 °C were investigated. However, there has been limited attention to systematically determine titanium particle velocity, within the whole-flow-field of cold spray jet, under different processing conditions.

This study aims to characterize a titanium cold spray free jet under different propellant gas conditions with respect to stagnation temperature and pressure. The cold spray free jet was chosen to prevent complexities that may arise from the interaction of the supersonic jet with the substrate that leads to formation of bow shocks and turbulence (Ref 7). The two-dimensional titanium particle velocity profiles along the center plane of the free jet are determined in respect to spray propellant gas, nozzle stagnation temperature, and spray pressure. The results show that PIV measurement successfully quantifies the velocity of titanium particles in a supersonic jet. Helium compared with nitrogen as a propellant gas presents a significantly larger supersonic flow field. An increase in temperature enhances particle velocity along the nozzle axis at the expense of in-flight particles at the edge of the supersonic jet. In contrast, an increase in pressure results

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in a more uniform increase in particle velocity within the jet. These experimental results prove that PIV is a useful characterization technique for optimization of cold spray parameters and validation of supersonic jet models.

2. Experimental Procedure

A commercial purity (CP) titanium, grade 4, powder with the composition presented in Table 1 was used for this study. The average particle size was 27 μm with powder size distribution and particle morphology as shown in Fig. 1. A cold spray system (CGT™ KINETIKS® 4000) was utilized, in which gas at elevated pressure was introduced to a heater and powder feeding vessel (Fig. 2). The high pressure and high temperature gas was taken into a converging/diverging (de Laval) nozzle. Supersonic flow conditions were achieved through compression of the gas through the nozzle throat followed by expansion to atmospheric pressure. The system incorporates two probes that measure the temperature and pressure before the converging section (stagnation area) of the nozzle. Two types of propellant gas, helium and nitrogen, were used with experimental conditions shown in Table 2. Titanium powder was fed into the stagnation region of the nozzle at 12 g min^{-1} feed rate. The nozzle geometry details are presented in Table 3.

Table 1 Composition of CP titanium powder

Ti	C, %	Fe, ppm	H, %	N, %	O, %	Si, %	Other, %
Balance	0.12	160	0.03	0.01	0.35	0.9	0.4 max

2.1 Particle Image Velocimetry Measurements

The PIV is a whole-flow-field characterization technique. The PIV systems measure velocity by determining particle displacement over a short separation time using a double-pulsed laser. A laser light sheet illuminates a plane

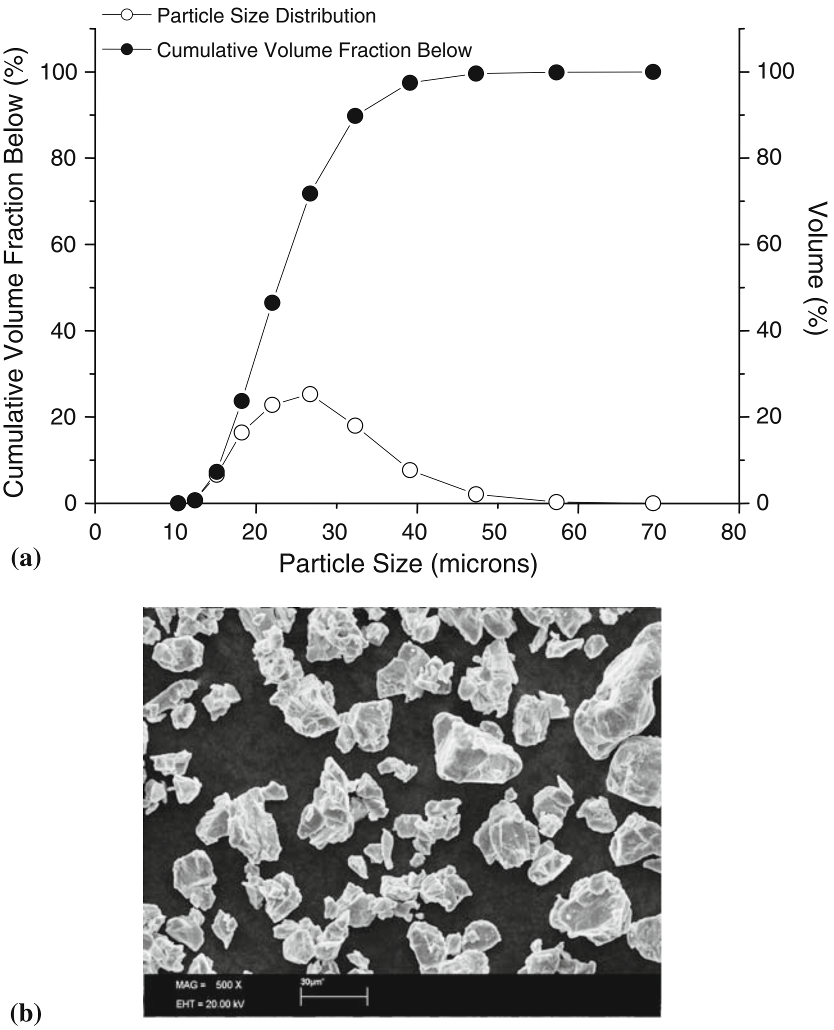

Fig. 1 (a) Size distribution and (b) morphology of the CP titanium powder

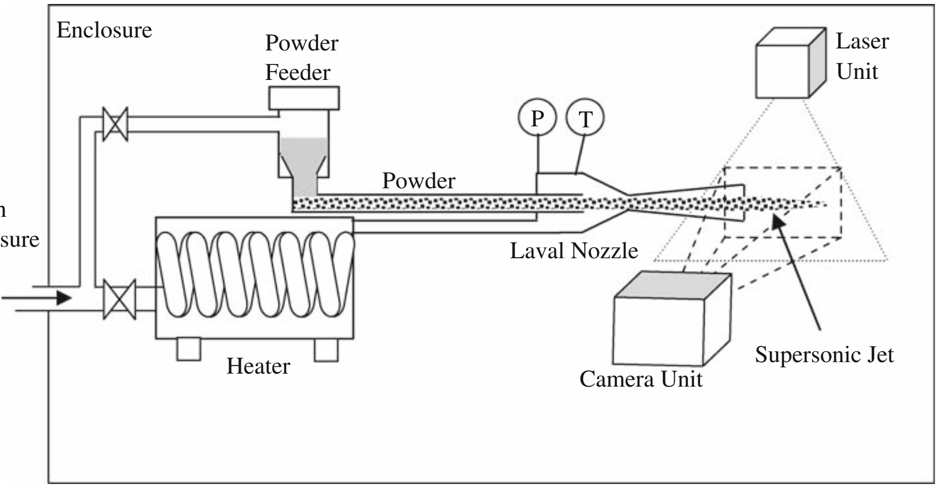

Fig. 2 Schematic representation of cold spray system and particle image velocimetry set up

Table 2 Experimental conditions used for PIV measurement of titanium cold spray free jet

Condition	T, °C	P, MPa	Propellant gas	PIV measured maximum velocity, m s^{-1}
1	550	1.4	Helium	900
2	550	1.4	Nitrogen	640
3	550	2.5	Nitrogen	730
4	750	2.5	Nitrogen	790

Table 3 Nozzles geometries and corresponding expansion ratio

	Throat diameter, mm	Exit diameter, mm	Divergent section length, mm
Nozzle	2.6	8.5	71.3

in the flow, and the positions of particles in that plane are recorded using a digital camera (Fig. 2). A fraction of a second later, another laser pulse illuminates the same plane, creating a second particle image. From these two subsequent particle images, PIV analysis algorithms obtain the particle displacements and velocity information for the entire flow region imaged.

In the current study, an ILA 2D PIV system was used, consisting of a SensiCam 12-bit digital CCD camera (1280 × 1024 pixels) synchronized with a New Wave 120 mJ double-cavity Nd:YAG Laser. The output laser beam with 4 ns pulse duration at wavelength of 532 nm is guided through an articulated arm system to the measurement location, where the beam is expanded by a cylindrical lens to form a 1.5- to 2-mm thick planar vertical light sheet over the measurement plane. The typical field of view of the CCD camera is 278 × 222 mm^2 using 1280 × 1024 pixels of CCD array. The smallest resolvable length is 217 μm, which is the real length of each pixel. The interrogation windows are 16 × 16 pixels (3.5 × 3.5 mm^2), with 50% overlap between consecutive interrogation cells, providing a velocity vector spacing of 8 pixels (1.75 mm). The laser pulse separation time was set to minimum at 4 μs in order to capture the maximum particle velocity up to 900 m/s. The uncertainty of pixel displacement due to calibration errors and image aberrations in the current study is approximately 0.2 pixels within the interrogation windows of 16 × 16 pixels. In each PIV measurement, 800 pairs of successive images were taken at the laser repetition rate of 4 Hz. Hence, a mean velocity flow pattern was obtained by statistically averaging 800 successive instantaneous velocity vector maps over approximately 3 min.

3. Results

The experimentally determined velocity using PIV reveals the velocity fields after particles depart from nozzle exit. Morphology of the supersonic flow flied (i.e., jet length and jet width) may provide an estimate on the distance between the nozzle exit and substrate, known as ‘stand off’. The optimized location for stand off distance is generally where the highest velocity for particles is reached outside of the nozzle. The maximum velocity achieved outside of the nozzle must be sufficient for deposition of particles. Therefore, a minimum velocity known as ‘critical velocity’ exists for deposition. Determination of critical velocity is complicated because of complicated thermomechanical events between particle and substrate under shock load and at the moment of impact. The critical velocity 650 m. s^{-1}, reported in previous studies (Ref 13), is considered to be adequate for examination of titanium PIV measurements in this study.

3.1 Effect of Propellant Gas

The two-dimensional (2D) velocity results outside the nozzle for helium at 550 °C and 1.4 MPa are shown in Fig. 3(a). It is worth noting that there is a slight upwards

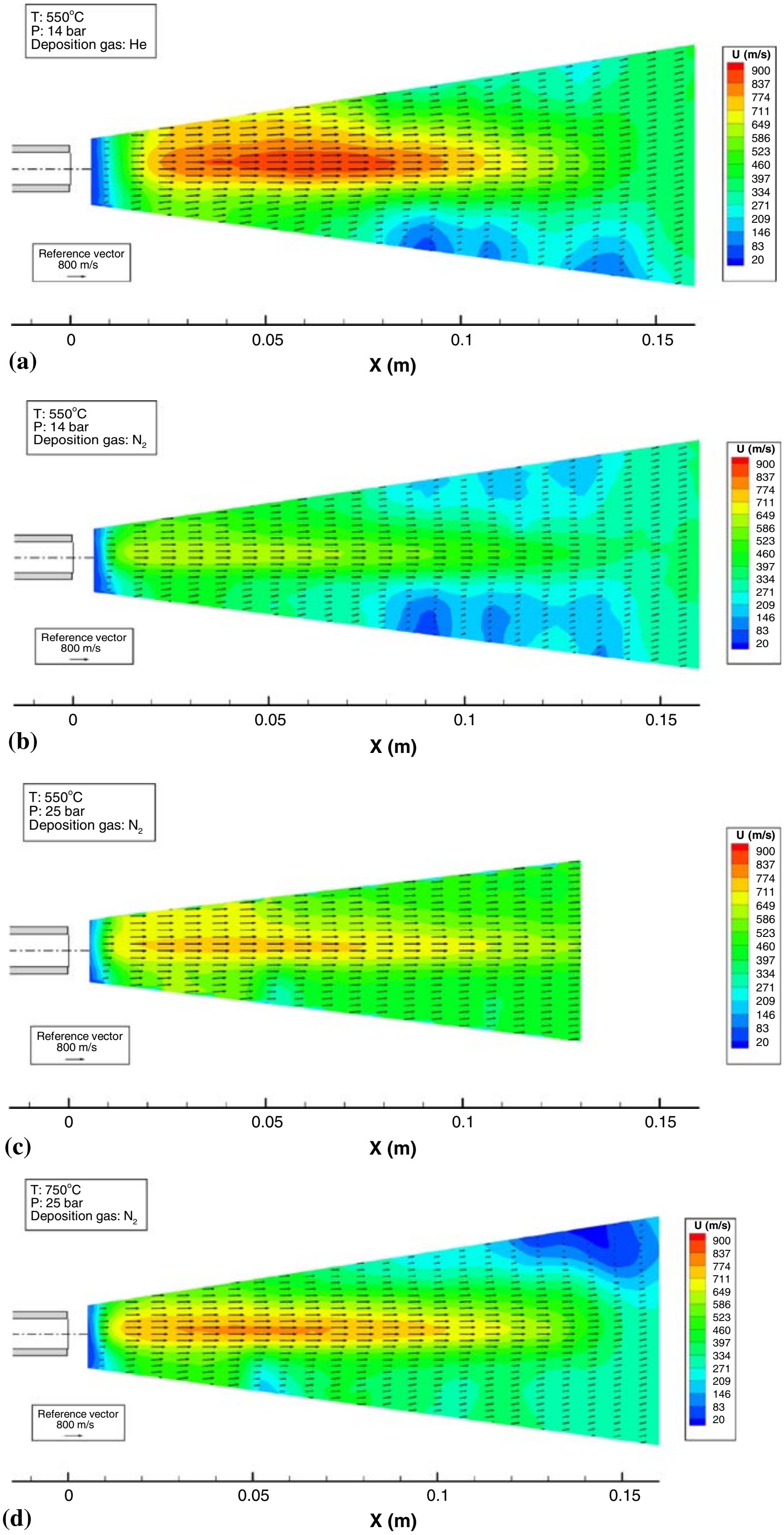

Fig. 3 PIV representation of instantaneous velocity vector field and average scalar velocity map for cold spray CP titanium supersonic jet with (a) helium at 550 °C and 1.4 MPa (b) nitrogen at 550 °C and 1.4 MPa (c) nitrogen at 550 °C and 2.5 MPa (d) nitrogen at 750 °C and 2.5 MPa

shift of the jet from the axis of the nozzle which is due to the powerful extraction system in cold spray enclosure. The supersonic jet length outside of the nozzle for particles that reach velocities above 650 m s^{-1} is 125 mm. The highest velocity region extends 80 mm away from the nozzle exit and includes particles that reach 900 m s^{-1}. This high velocity region in the jet indicates extra kinetic energy available for extensive deformation of particles which is required for a dense titanium deposit. These observations confirm the previous finding by Li & Li (Ref 14) regarding the high velocity achieved due to high speed of sound in helium.

Figure 3(a) represents that the helium jet for particles that reach velocities above 650 m s^{-1} is wider than 8.5 mm (the nozzle exit diameter). This suggests that, under deposition conditions, a large proportion of particles in the jet reach critical velocity resulting in improved deposition efficiency as reported in earlier investigations (Ref 12, 14). In contrast to helium, titanium particles at 550 °C and 1.4 MPa under nitrogen attain significantly lower velocity. The high velocity region of supersonic flow field for nitrogen compared with helium is considerably decreased (Fig. 3a and b). The highest velocity region corresponds to particles with velocities below 650 m s^{-1} with a jet length of 70 mm. Indeed the maximum velocity determined for this condition was 640 m s^{-1}. This suggests that under this condition titanium particles have insufficient velocity for deposition and significantly lower deposition efficiency is expected.

3.2 Effect of Pressure

An increase in pressure from 1.4 to 2.5 MPa, at 550 °C stagnation temperature, led to an increase in maximum velocity from 640 to 730 m s^{-1}. This is most likely related to an increase in flow of nitrogen from 33 to 60 m^3 h^{-1} with an increase in pressure from 1.4 to 2.5 MPa, respectively. The improved velocity at 2.5 MPa is expected to provide more kinetic energy for particles in-flight and further expansion of the jet outside of the nozzle. For instance, an increase in pressure from 1.4 to 2.5 MPa lead to an increase in jet length from 70 to 110 mm for particles that reach 590 m s^{-1} in the jet (Fig. 3b and c). Further to this, the flow field results reveal that the effect of shock waves which may cause an abrupt change in particles velocity was insignificant for the chosen conditions.

3.3 Effect of Temperature

Figure 3(c) shows PIV of titanium supersonic flow at 550 °C and 2.5 MPa with nitrogen as propellant gas. The jet length for the highest velocity region, 711-774 m s^{-1}, was 75 mm. The maximum velocity outside of nozzle was 730 m s^{-1}, which is 80 m s^{-1} more than the required velocity 650 m s^{-1} for deposition of titanium and therefore high deposition efficiency is expected. For a similar velocity range, 711-774 m s^{-1}, an increase in stagnation temperature from 550 to 750 °C results in an extension of the jet length from 75 to 100 mm, respectively (Fig. 3c and d). The maximum velocity for 750 °C stagnation temperature was 790 m s^{-1} that is 60 m s^{-1} more than 550 °C condition. These PIV results are in agreement with previous (Ref 11, 14) reports that suggest an increase in stagnation temperature leads to increased velocity for cold spray particles.

4. Discussion

The PIV results reveal 2D velocity flow field of titanium particles in a cold spray supersonic jet. This velocity distribution provides valuable information on the way in which a cold spray nozzle performs under different processing conditions. The optimum distance between the nozzle exit and substrate (stand off) is only achieved by knowledge of velocity distribution outside of nozzle. For example, it is generally accepted that the place at which maximum particle velocity is achieved is the most appropriate location to position the substrate (Ref 12). This location is chosen to exploit the highest kinetic energy available for successful deposition. For this reason, the PIV results were analyzed to determine the relationship between the supersonic flow field and the location at which the maximum velocity for titanium particles is achieved outside of the nozzle.

4.1 Effect of Distance from Nozzle Exit

The velocity determined by PIV as a function of distance at 550 °C and 1.4 MPa with helium as propellant gas is shown in Fig. 4. It is evident that titanium particles along the axis of nozzle continue to accelerate outside of the nozzle until a maximum velocity of 900 m s^{-1} is reached 60 mm from nozzle exit. Pattison et al. (Ref 12) have observed a similar trend and related this to density (4.5 kg/m^3) and inertia of titanium particles which leads to slower acceleration within the nozzle allowing for further increase in speed after particles depart from nozzle. Moreover, these results indicate that a distance shorter than 60 mm from the nozzle may compromise particle velocity and optimum deposition conditions.

Further increase in distance beyond 60 mm from nozzle exit leads to a decline in velocity. Considering 650 m s^{-1}, the critical velocity for deposition, it is expected for titanium particles to deposit successfully at 60 mm from nozzle exit. This, however, is noticeably different for a similar condition in which nitrogen is used. The maximum velocity 640 m s^{-1} was achieved 28 mm from nozzle exit. This is 32 mm shorter than helium condition (Fig. 4). However, at 550 °C an increase in cold spray pressure from 1.4 to 2.5 MPa, increases the distance from 28 to 42 mm (Fig. 4). This suggests that a larger stand off must be considered if higher pressure (gas flow) is going to be used for deposition. This, however, is less critical for temperature. For example, an increase in temperature from 550 to 750 °C led to only 3 mm increase in the location where maximum velocity reached from 42 to 45 mm (Fig. 4). Overall, the PIV results confirm that

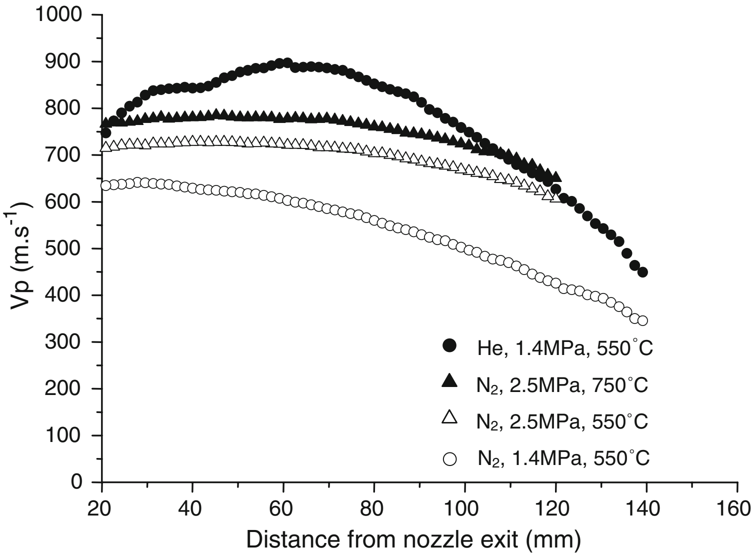

Fig. 4 Comparison of CP titanium particle velocity profile, in respect to the cold spray nozzle axis, as a function of distance from nozzle exit for all conditions in Table 2

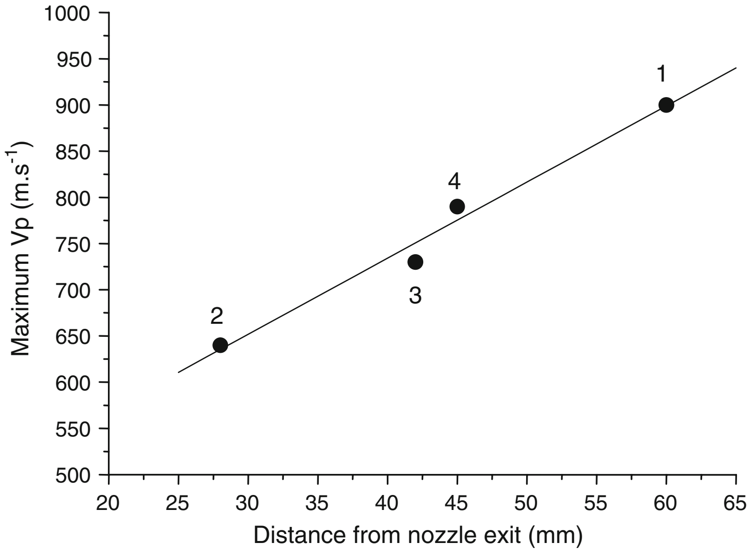

Fig. 5 The maximum titanium particle velocity outside of the cold spray nozzle as a function of distance from the nozzle exit (numbers on the graph represent experimental conditions in Table 2)

helium and spray pressure have more profound effect than stagnation temperature on acceleration of particles outside of nozzle. This improved acceleration pushes the critical location at which particles reach maximum velocity further away from nozzle exit as summarized in Fig. 5 for conditions in Table 2.

4.2 Velocity Distribution Normal to Jet Direction

Identification of the place at which particles achieve maximum velocity within the supersonic jet is important but insufficient for optimization of cold spray process. This is because of the particle velocity profile normal to the jet direction. For example, Fig. 6 shows the measured particle velocities profile for the helium condition. The maximum velocity 900 m s^{-1} was achieved 60 mm from the nozzle exit. The velocity profile was established with reference to nozzle diameter which was 12 mm. It is worth noting that the velocity distribution in Fig. 6 is not symmetrical with respect to nozzle centerline because of the slight shift of the supersonic jet previously observed in Fig. 3. It is obvious from Fig. 3 that this effect does not influence the overall shape of the spray plume. However, these results reiterate the importance of PIV

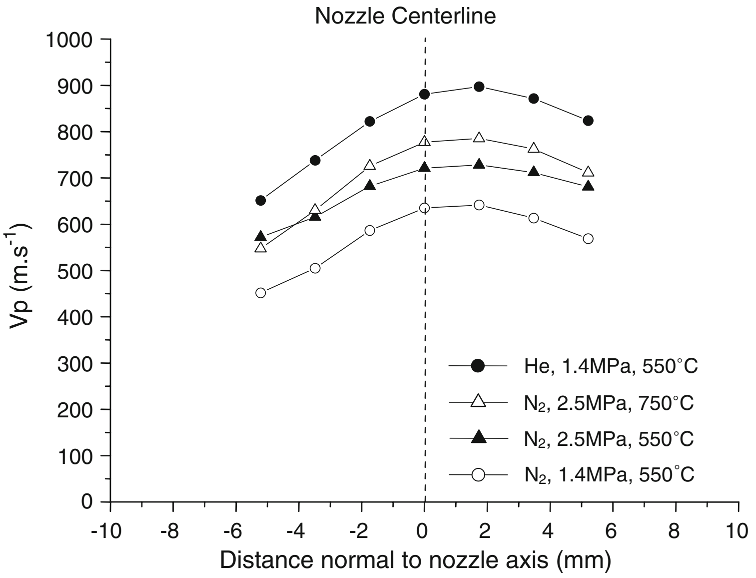

Fig. 6 Measured velocity profile normal to cold spray nozzle centerline for all experimental conditions in Table 2 where maximum velocity was achieved outside of the nozzle

measurements for determination of real jet conditions that are extremely difficult to anticipate using current modeling approaches.

The lowest velocity 650 m s^{-1} for helium condition is achieved when in-flight particles are located 5 mm away from the nozzle centerline (Fig. 6). This is the critical velocity for deposition of titanium and it is expected for particles to deposit with very limited deformation due to insufficient velocity and kinetic energy. In contrast, at the locations close to nozzle axis 900 m s^{-1} velocity is reached that is 250 m s^{-1} above the critical velocity. This extra velocity provides the kinetic energy for particles to deform as splats and create improved bonding. These observations confirm that velocity profile in cold spray jet, however, may lead to inhomogeneous particle bonding within the cold spray titanium deposit which is not desirable for improved mechanical properties. Hence, for improved homogeneity in deposited material, it seems logical to optimize cold spray parameters in such a way that a more uniform velocity normal to the nozzle axis is achieved.

Figure 6 shows that nitrogen, instead of helium, at 1.4 MPa accelerates titanium particles to maximum 400 m s^{-1}. This velocity is reached 5 mm from nozzle centerline. It is interesting to note that the difference between the maximum and minimum values for velocity under nitrogen is 240 m s^{-1} that is almost the same as helium condition with 250 m s^{-1}. However, Fig. 6 shows a decline in the difference between the highest velocity and the lowest velocity from 240 to 160 m s^{-1} when pressure was increased from 1.4 to 2.5 MPa. This suggests that nitrogen supersonic jet at 550 °C, 2.5 MPa provides a more uniform particle velocity which may improve homogeneity in deposited material. However, an increase in stagnation temperature from 550 to 750 °C exacerbates the uneven distribution of velocity accelerating particles at the vicinity of the nozzle axis to 790 m s^{-1} and decelerating in-flight particles further away from the nozzle centerline to minimum 550 m s^{-1} (Fig. 6). This means that an increase in temperature results in particle acceleration at the core of the jet at the expense of velocity for in-flight particles located at the edge of the supersonic jet. The consequence of this is most likely uneven deposition of titanium. This, however, is worth considering because it is generally accepted that an increase in stagnation temperature improves particle velocity. This, however, may contribute to a non-uniform titanium deposit with implications such as weak particle-particle and particle-substrate bonding. Further study is required to examine the effect of velocity profile on titanium deposit microstructure.

The normalized velocity values in Fig. 7 summarize the relative impact of spray parameters on distribution of velocity normal to the nozzle axis. This corresponds to the distance from the nozzle exit where the maximum velocity for particles is reached. A decrease in cold spray pressure from 2.5 to 1.4 MPa at 550 °C results in a decrease in velocity of in-flight particles at the edge of the jet. A similar trend is observed when stagnation temperature is increased from 550 to 750 °C with a limited influence of helium as propellant gas. These PIV profiles confirm complexities that arise from the interaction of titanium particles with supersonic propellant gas. Further to this, PIV provides valuable information about cold spray nozzle performance in respect to deposition parameters. Perhaps the most challenging optimization for cold spray process is minimization of velocity distribution normal to the nozzle axis where maximum particle velocity is reached outside of nozzle. This is to uniformly deposit titanium that is critical for improved mechanical properties of the cold spray coating and fabrication process.

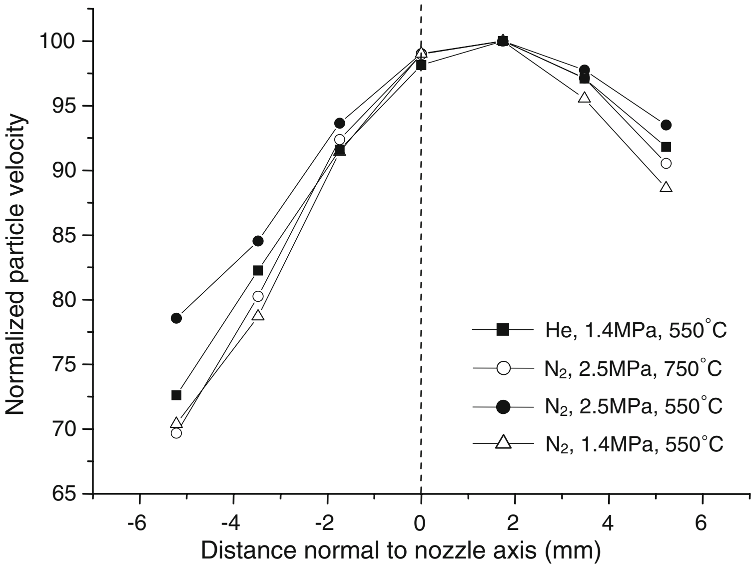

Fig. 7 Normalized velocity profile normal to cold spray nozzle axis for all experimental conditions in Table 2 where maximum velocity was achieved

5. Conclusions

The velocity profile for cold spray supersonic jet was determined experimentally using PIV. Results confirmed that titanium particles accelerate outside of the nozzle particularly when helium is used as propellant gas. Helium compared with nitrogen significantly extends the high velocity flow region outside of the cold spray nozzle. An increase in particle velocity increases the distance between the nozzle exit and the location at which maximum velocity is reached. An increase in stagnation temperature mostly contributes to acceleration of in-flight particles at the vicinity of the nozzle axis. In contrast to temperature, an increase in pressure expands the high velocity regions normal to the nozzle centerline. The outcomes of PIV technique proved to be useful for optimization of cold spray parameters and validation of the future models for this process.

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